ADSORPTION OF TOXICANTS (CHROMIUM, IRON AND OXALIC ACID) ON ACTIVATED CARBONS PREPARED FROM TAMARIND SEEDS

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ABSTRACT: **Activated carbon prepared from tamarind seeds is used to adsorb selected heavy metals such as chromium and Iron ions and oxalic acid and the adsorption capacity of activated carbon from tamarind seed was compared with the adsorption capacity of commercially available activated charcoal.Tamarind seeds which is an agricultural wastes and also waste product in many industries was activated with sulphuric acid and then used for the removal of these toxic metals from their aqueous solutions. The effects of varying the adsorbent dosage, contact time, pH of the solution mixture of adsorbent and toxic material on the adsorption capacity of the activated carbons were determined. The characterization of activated carbon was analysed by FTIR. The batch experiments were conducted for different adsorbent dosage, contact time and different pH. Adsorbent dosage of 2.5 g gave the Maximum adsorption for all the three toxic materials (chromium, Iron and oxalic acid).Adsorption of materials increases with increase in the adsorbent dosage. At lower pH, more adsorption of metals was observed. Maximum adsorption took place in the pH range of 3-5. With increase in the agitation time or contact time, the adsorption of metals also increased. Optimum adsorption occurred at 5 hr contact time. Adsorption studies are carried out with commercial activated carbon and are compared with the results obtained from tamarind seed activated carbon. But considering the overall economics activated carbon from tamarind seed gives better results of adsorption.**

Keywords: **Adsorption, Activated carbon, Tamarind seeds, Parameters, Adsorption Experiments, Adsorption Isotherms.**

1.0.INTRODUCTION

Now-a-days environmental pollution is the one of the biggest issue in many countries. Environmental pollution may be in the form of air pollution, water pollution and others. Water pollution contributes the major environmental pollution in several countries which shows great impact on human life and biodiversity. The important sources of water pollution are industrial effluents and domestic sewage. General contaminants in effluent water are heavy metals, phenols, dyes, toxic organics, ligands, phosphorous and suspended solids[1-2]. Treatment of effluent water plays an important role in many industries. Various process like adsorption, flocculation, ultrafiltration, ion exchange, electrochemical process, coagulation are used to treat effluent water treatment out of which adsorption proved to be most well known and economical process for treatment and renovation of waste water. The ability of certain solids to preferentially concentrate specific substances on their surfaces is the basic principle for adsorption. In addition to the conventional adsorbents, Many lingo -cellulosic materials including coirpith, stalks rice, barley husk, peanuts, coconut shell, cashew nut shell, tamarind seeds are used as adsorbents. Active carbons are obtained by either chemical activation or physical activation of precursor in chemical activation KOH, H_2SO_4, H_3PO_4, $ZNCL_2$, K_2CO_3, NA_2CO_3 are used as activating agents [3-4]. Tamarindusindica (tamarind) is a fruity species which is more available in India, Mayanmar, Phillipines and Indonesia. It is well known for its adaptability to various climatic and edaphic conditions and large production. Tamarind fruit consist of pulp and hard seeds. Seeds contribute 30-40% of fruit which goes waste in many processes like cooking, soups, preparation of beverages[5]. Tamarind seed has also been used as a raw material for granular activated carbon prepared by incineration and was activated by H_2SO_4 at 150˚C. Pollution of natural water resources by iron is one of the most important problems that face and threaten the world. Fe contaminants are producedfrom liquid wastes discharged from a number of industries [6]. Water with high iron content can be very objectionable in taste, odour, or appearance. With severe iron poisoning, much of the damage to the gastrointestinal tract and liver may be the result of highly localized iron concentration and free radical production leading to hepatotoxicity through lipid peroxidation and the destruction of the hepatic mitochondria.With this result, the liver becomes cirrhotic. Hepatoma, the primary cancer of the liver, has become the most common cause of death among patients with hemochromatosis [7]. Activated carbon has been proved to be an excellent adsorbent for removing organic or inorganic pollutants[8]. Chromium is omni present in the environment, ranking 21[M] among the elements in crystal abundance. It is found in varying concentrations in air, soil, water and all biological matter. It is abundant in earth's crust. The primary anthropogenic sources of chromium include domestic wastewater, manufacturing processes involving metals and the dumping of sewage sludge. Total chromium is generally detected at low conentrations in fresh water. The oxidation stale of chromium is very important relative to its mobility and its role in plant and human nutrition. The hexavalent form, Cr(VI), is more toxic and also known to be more mobile in the soil environment than Cr(III). Cr(III) is classified as a hard acid and forms relatively strong complexes with oxygen donor ligands. Cr(VI) is water soluble, always existing in solution as a component of complex anion. Chromium is an essential trace element forming a part of the anti-diabetogenic factor, which is essential for the metabolism of insulin[9,10].Oxalic acid is an organic ligand which is a important pollutant in industrial waste. High levels of oxalic acid causes health problems like kidney stones,uremia,erosion of enamel and mouth, vomiting and heamatemesis[11].Activated carbon has been proved to be an excellent adsorbent for removing organic or inorganic pollutants. It could be produced from agricultural byproducts with low cost and abundance [12,13].In this research, tamarind seed was used as precursor for the production of activated carbon using KOH activation.The effects of KOH : tamarind seed ratios and activation temperature were studied. The physical and chemical properties were characterized. Finally, the activated carbon prepared in this study was used in preliminary test for Cr^{6+}, Fe^{2+},oxalic acid adsorption from aqueous solution.

2.0.MATERIALS AND METHODS

2.1.SAMPLE COLLECTION:

Samples of tamarind seeds were collected from local Market in anantapur, Andhra Pradesh State. Tamarind seeds were washed thoroughly with tap water and then with distilled water to remove all trace of impurities, oil, dirt, dust and salts. The samples were sundried and then dried in a hot air oven at 130°C for 3 hours.

Fig 2.1: Raw tamarind seeds

2.2.PREPARATION OF ADSORBENT (ACTIVATED CARBON FROM TAMARIND SEEDS):

The dried seeds were crused into smaller particle sizes by using a mixer. The particles were sieved into fine powder of diameter 0.177mm using a 85 BSS mesh. The fine powder of tamarind seed was activated by treating with sulphuric acid (98%w/w) in 1:4 weight ratio and then vaccum filtered. The mixture of powder and sulphuric acid was then dried in oven at 150°C for 24 hours. The carbonized material was then washed with distilled water to remove free acids and then dried in hot air oven at 150°C for 3 hours to form activated carbon.

Fig 2.2: Tamarind seed powder

Fig 2.3: Activated carbon from tamarind seeds

2.3.Characterization Studies:

2.3.1.pH Determination:

Solution of the activated sample of tamarind seed powder of 1% (w/v) was prepared using mixture of the sample in deionized water. pH of the supernatant was obtained after 2hrs by using pH indicator strips/papers[14].

2.3.2.Bulk density :

This was determined by measuring the volume of water displaced when a known weight(2.00 g) of sample of activated carbon were dropped into a graduated measuring cylinder with tapping until the activated carbon occupied a initial volume. A 100 cm^3calibrated measuring cylinders was washed and dried. 50 $cm^3$of the water was added to the measuring cylinder and was noted. 2 grams of the sample was transferred into the measuring cylinder and volume of the increased water level was recorded. Care was taken to ensure there were no air bubbles before taking volume measurements.

2.3.3.Density of Carbonized Material :

The oven dried tamarind seed powder(activated carbon) of a known weight Wa (2.0 g) was put into a measuring cylinder, was tapper gently and volume occupied was recorded as Va. A known volume of distilled water was added slowly through the side of the cylinder to soak the sample thoroughly. The final volume containing activated carbon and distilled water was recorded as Vb.

The particle density is calculated as: $P\ (g/cm^3) = Wa/(Vb-Va)$

2.4.FTIR analysis:

This was carried out on the activated carbon from tamarind seeds.This technique was used to determine the functional groups in the adsorbent which are responsible for adsorption of the selected metals. The FTIR was in the range 4000 - 400 cm-1 region with resolution of 4 cm-1. For this analysis a small amount of granulated sample of tamarind seed activated carbon was encapsulated in a sample holder[15]. The FTIR was recorded on a Fourier Transform Spectrometer at the Raghavendra Institute of Pharmaceutical Education and Research(RIPER),Anantapur,AP,India.The scans were collected in the spectral range specified above.

Fig 4: FTIR Spectrophotometer

2.5. Adsorption experiments:

Exactly 30.0cm^3 of stock solution was measured into 250 cm^3 conical flask and known weight of adsorbent was transferred into the conical flask. The mixture was agitated in a shaking incubator at 120 rpm for known time and then taken out from shaker. Then the mixture was filtered through a whatman filter paper into another 250cm^3 conical flask. The concentration of the residual or remaining metal ion in the filtrate after adsorption process was determined using titration procedure[16-17].

The amount of the metal adsorbed was calculated using the equation

$qe = V(Co - Ce)/1000m$

where,

qe is the amount of adsorbate ion adsorbed in milligram per gram of the adsorbent.

Co is the initial concentration of the metal ion before the adsorption process.

Ce is the equilibrium concentration of the metal ion in the filtrate after adsorption process.

m is the mass of the adsorbent.

V is the volume of the solution in cm^3.

The equation that gives percentage of metal ion removal by the plantain peel adsorbent is given by:

$\%\ Adsorbed = ((Co - Ce)/Co) \times 100$

Where,

Co is the initial concentration of the metal ion before the adsorption process.

Ce is the equilibrium concentration of the metal ion in the filtrate after adsorption process.

Fig 5: Shaking Incubator

2.5.1.Effect of Adsorbent Dosage on the Removal of toxicants (Chromium, Iron and oxalic acid):

Adsorbent dosage of 0.5, 1.0, 1.5, 2.0 and 2.5 g were used to determine the adsorption efficiency of activated carbon of tamarind seed on these selected metals. Each of the pre-weighed adsorbents were mixed with 100 mg/cm^{3}of selected heavy metals, agitated for predetermined time in a Shaking incubator at 30˚C and 120 rpm. The adsorbent and adsorbate solution were separated by filtration using whatman filter paper. The concentrations of un-adsorbed heavy metals were determined using titration procedure[18,19].

2.5.2.Effect of contact time on the Removal of toxicants (Chromium, Iron and oxalic acid):

Different contact times were maintained for different adsorbent dosage of 0.5, 1.0, 1.5, 2.0 and 2.5 g and the adsorption efficiency of activated carbon of tamarind seed on these selected metals was determined. Each of the pre-weighed adsorbents were mixed with 100 mg/cm^{3}of selected heavy metals, agitated for different contact times in a Shaking incubator at a agitation speed of 120 rpm and maintained at 30˚C . The mixture of adsorbent and adsorbate solution was separated by filtration using whatman filter paper. The concentrations of un-adsorbed heavy metals were determined using general titration procedure [18,19].

2.5.3. Effect of pH on the Removal of toxicants (Chromium, Iron and oxalic acid):

PH of mixture of adsorbent and adsorbate solution was maintained by using 0.1 N HCL and 0.1 N NaOH with the help of pH strips. Adsorbent dosage of 2 g was used to determine the adsorption efficiency of activated carbon of tamarind seed on these selected metals. Adsorbent dosage of 2 g was mixed with 100 mg/cm^{3}of selected heavy metals, agitated for a contact time of 3 hrs in a Shaking incubator at a agitation speed of 120 rpm and maintained at 30˚C. The mixture of adsorbent and adsorbate solution was separated by filtration using whatman filter paper. general titration procedure was used to determine the concentrations of un-adsorbed heavy metals[18,19].

2.5.4. Comparison of adsorption of toxicants (Chromium, Iron and oxalic acid)with commercial activated carbon:
Exactly 30.0cm^3 of chromium solution was measured into 250 cm^3 conical flask and known weight of commercial activated carbon was transferred into these conical flask. The mixture was agitated in a shaking incubator at 120 rpm for 2 hr of time and then taken out from shaker. Then the mixture was filtered through a whatmann filter paper into another 250cm^3 conical flask. The concentration of the residual or remaining metal ion in the filtrate after adsorption process was determined using titration procedure. These results were compared with the results obtained by conducting the same experiment for chromium stock solution with the activated carbon prepared from tamarind seeds.

2.5.5. Adsorption Modelling of Heavy Metal ions:

Batch equilibrium adsorption experiments have been carried out to determine the adsorption capacity of the adsorbent. These adsorption experiments were performed in a conical flask (250 ml) where adsorbent with different dosages were mixed with 30 ml of stock solutions with initial concentration of 100 mg/cm^3. The pH was adjusted to 7 by adding either few drops of diluted hydrochloric acid or sodium hydroxide. These mixure was agitated in a shaking incubator at a agitation speed of 120 rpm and at a temperature of 30°C for different contact periods. Thereafter, the different supernatants were filtered and the concentration remaining in the filtrates were determined using general titration procedures. Adsorption rates of different samples were determined from the level of the selected heavy metals in their aqueous solution. Langmuir Isotherm model and Freundlich Isotherm model were used to determine the degree of adsorption[20,21,22].

3.0. RESULTS AND DISCUSSION

3.1.Physicochemical Properties:
The results of determinations on activated carbon prepared from tamarind seeds are presented in the table 1 below:
Table 3.1: Physicochemical Properties of activated carbon prepared from tamarind seeds

Parameter	Value
Bulk density (g/cm^3)	0.4
Density of Carbonized Material (g/cm^3)	2.5
Moisture Content (%)	2.9704
Ash Content (%)	1.551035
Dry matter	33.6762
pH	7.0

3.2.Characterization of the Adsorbent:

3.2.1Fourier Transform Infrared Analysis (FTIR Analysis):

The Functional groups in the activated carbon from tamarind seeds are given by FTIR spectra and listed out in Table 2. The FTIR spectra obtained for the activated carbon prepared from tamarind seeds samples is given in following fig 1;

Table 3.2: FTIR showing the different bonds and functional groups present in the tamarind activated carbon

S.NO.	Frequency,cm^{-1}	Bond	Intensity	Functional group
1	614.37	-C≡C-H; C-H bend	Very sharp	Alkynes
2	653.99	=C-H bend	sharp	Alkenes
3	1047.28	C-N stretch	strong	Aliphatic amines
4	1162.15	C-H wag ($-CH_2X$)	strong	Alkyl halides
5	1335.76	C-N stretch	Weak	Aromatic amines
6	1540.33	N-O asymmetric stretch	strong	Nitro compounds
7	1618.85	C-C stretch (in-ring)	Weak	Aromatics
8	1696.90	C=O stretch	strong	α-β unsaturated aldehydes, ketones
9	1736.91	C=O stretch	Weak	Ketones, saturated aliphatic
10	2204.09	-C≡C- stretch	Weak	Alkynes
11	2623.44	O-H stretch	strong	Carboxylic acids
12	2746.73	H-C=O; C-H stretch	weak	Aldehydes
13	2919.75	C-H stretch	strong	Alkanes
14	3351.57	N-H stretch	weak	1˚2˚ amines ,amides
15	3481.64	O-H stretch, H-bonded	weak	Alcohols,Phenols

3.2.2.Effect of Adsorbent Dosage on the Removal of Toxicants (Chromium, Iron and oxalic acid):

The effect of adsorbent dosage on Iron ions, oxalic acid and chromium ions are shown in fig 2, fig 3,fig 4 respectively. The solutions with adsorbents with 0.5, 1, 1.5,2 ,2.5 g adsorbent dosages were shaken at constant temperature of 34˚C in a shaking incubator with 120 rpm speed.

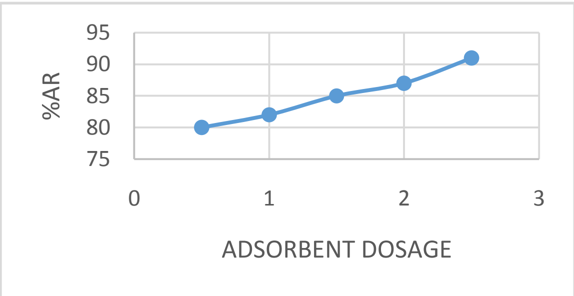

Fig 3.1: Effect of adsorbent dosage on Iron

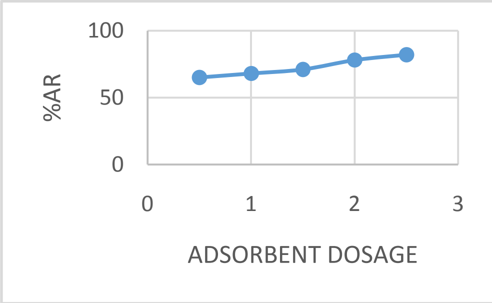

Fig 3.2: Effect of adsorbent dosage on Oxalic acid

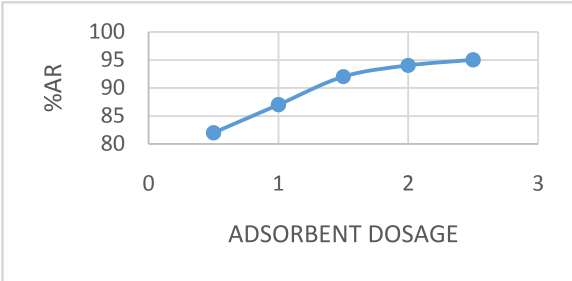

Fig 3.3: Effect of adsorbent dosage on Chromium Ions

The percent removals of metal ions were found to increase with an increase in the mass of adsorbent. At 2.5 g of adsorbent dosage, Maximum adsorption was observed for chromium with 95% adsorption, followed by Iron with 91% adsorption and minimum for oxalic acid with 82% adsorption.

3.2.3.Effect of contact time on the Removal of Toxicants (Chromium, Iron and oxalic acid):
The effect of contact time on chromium, Iron and oxalic acid are shown in fig 5; The solutions with adsorbent were shaken at constant temperature of 34˚C in a shaking incubator with 120 rpm with 2.5 g adsorbent dosage.

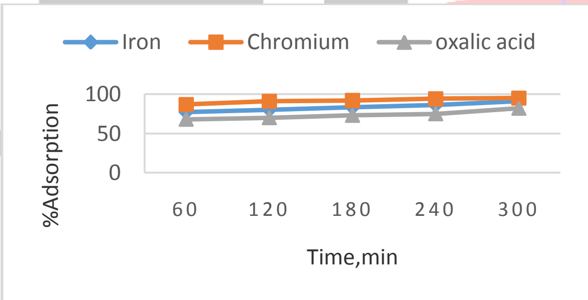

Fig 3.4:Effect of contact time on chromium, Iron and oxalic acid

Maximum percentage of 95% adsorption of chromium ions occurred at 5 hr and 91% of Fe^{2+} ions and 82% of oxalic acid at 5 hr. As contact time increases, the percentage adsorption increased. The Maximum adsorption occurred for chromium followed by Iron and then oxalic acid. The patterns of the curve established that the adsorption is time dependent in which adsorption capacity was proportionally related to the time. This is in agreement with the adsorption kinetic theory which stated that the higher the time, the more the amount of fluid is adsorbed on the adsorbent. Initially, there were large number of vacant active binding sites available at the first phase of experiment and large amount of metal ions were bound rapidly on activated carbon at a faster adsorption rate .The binding site shortly became limited and the remaining vacant surface sites were difficult to be occupied by metal ions due to the formation of repulsive forces between the metal ions on the solid surface and the liquid phase.

3.2.4.Effect of pH on the Removal of Toxicants (Chromium, Iron and oxalic acid):
The effect of pH on chromium, Iron and oxalic acid are shown in fig 6;The solutions with adsorbent were shaken at constant temperature of 34˚C in a shaking incubator with 120 rpm with 2 g adsorbent dosage.

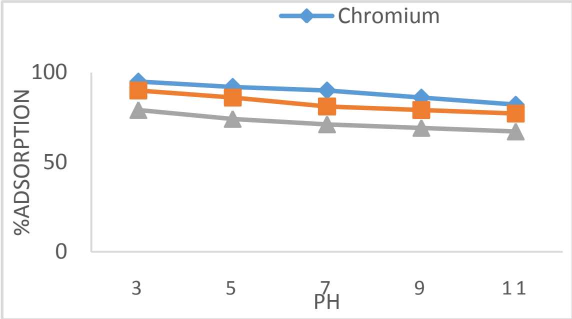

Fig 3.5:Effect of pH on chromium, Iron and oxalic acid

The pH is a controlling factor for many kinds of metal adsorption process from aqueous solution. The surface properties of adsorbents, ionic state of functional groups and species of metals are dependent on pH condition. It was observed that more adsorption removal occurred at lower pH. More adsorption was observed for chromium followed by Iron and then oxalic acid. At a pH of 3 Maximum adsorption occurred and the percentage removal was 95% for Cr^{6+}, 90% for Fe^{2+} Ions and 79% for oxalic acid for 2 g of adsorbent Dosage and a contact time of 3 hr.

3.2.5.Comparison of adsorption of toxicants with commercial activated carbon:

Comparison was made for the chromium metal as it showed maximum adsorption of the three toxicants. Commercial Activated carbon showed more adsorption than activated carbon from tamarind seeds. The difference in adsorption capacity between commercial activated carbon and tamarind seed activated carbon decreased with increase in adsorption dosage .At higher adsorbent Dosage activated carbon from tamarind seeds nearly approached Commercial Activated carbon in percentage removal. Comparison of adsorption removal was showed in the fig 7.

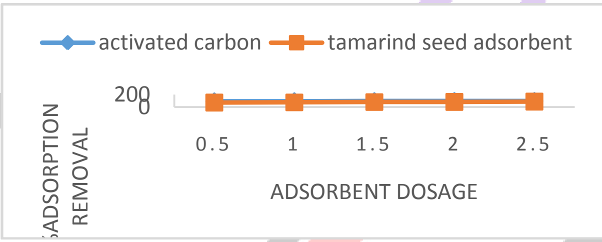

Fig3.6:Comparison of adsorption of toxicants with commercial activated carbon

3.2.6.Langmuir and Freundlich Isotherms Modeling for Metals Uptake:
The extent of adsorption capacity of selected metal ions were determined using Langmuir and Freundlich Isotherms Modeling as shown in Figures 8,9,10,11,12,13;

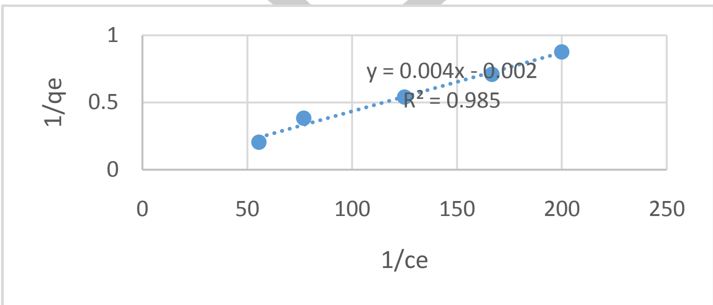

Fig 3.7: Langmuir Adsorption Isotherm for Chromium Adsorption

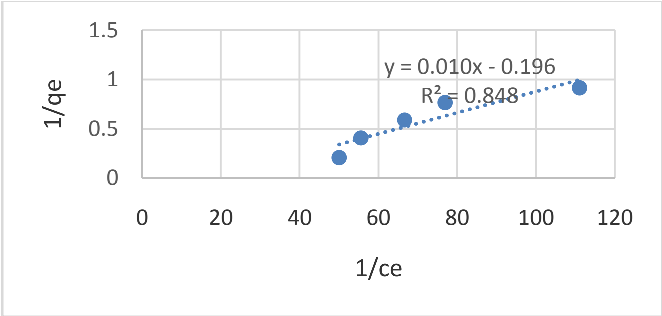

Fig3.8:Langmuir Adsorption Isotherm for Iron Adsorption

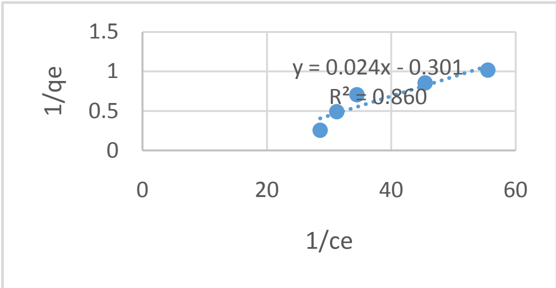

Fig 3.9: Langmuir Adsorption Isotherm for oxalic acid Adsorption

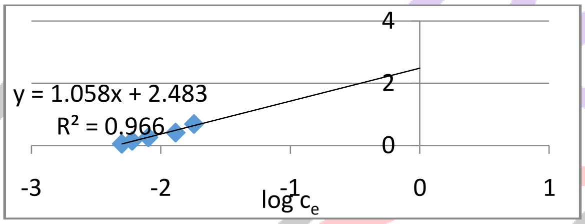

Fig3.10.:Freundlich Adsorption Isotherm for Chromium Adsorption

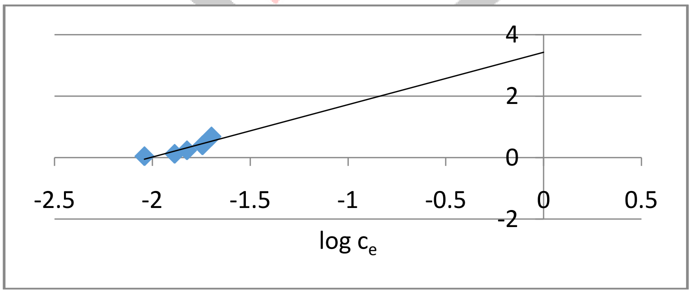

Fig 3.11: Freundlich Adsorption Isotherm for Iron Adsorption

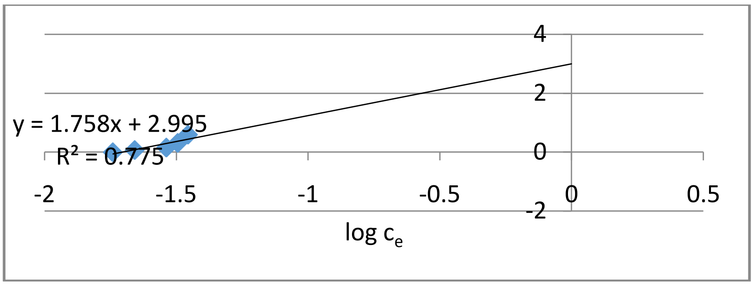

Fig 3.12: Freundlich Adsorption Isotherm for oxalic acid Adsorption

3.2.7.Adsorption Isotherm constants for adsorption of Chromium, Iron and Oxalic acid on activated carbon prepared from tamarind seeds:

Table 3.2:Adsorption Isotherm constants

Adsorption Model	Parameters	Cr^{6+}	Fe^{2+}	Oxalic acid
Langmuir Isotherm	q_{max}	40	5.0864	3.31895
	K_L	0.56818	18.3738	12.19838
	R^2	0.9852	0.8486	0.8603
Freundlich Isotherm	K_F	11.986	31.6424	19.9913
	1/n	1.058	1.7194	1.7587
	R^2	0.9668	0.814	0.7752

4.0.CONCLUSION:

Activated charcoal prepared from tamarind seeds are good alternative source for activated carbon production to commercial activated carbon. FTIR spectra analysis confirmed the presence of absorbance peaks of carboxyl, alkene, alkane, amide, aldehyde and alkyl functional groups. Thus, these functional groups are responsible for the adsorption of the selected metals.It can be used as an adsorbent for the adsorption of the cr^{6+}, Fe^{2+}, oxalic acid.The adsorption process is highly dependent on contact time, adsorbent dosage, pH. The adsorption capacity of activated carbon prepared from tamarind seeds with acid activators have comparable strength adsorption rate which nearly approaches the adsorption strength of commercial activated carbon. It was observed that the trend of metal affinity for activated tamarind seed surface was Cr^{6+}>Fe^{2+}>oxalic acid. Percentage of adsorption removal increased with increase in adsorbent dosage, increase in contact time and decrease in pH. Maximum adsorption of 95% was observed for chromium at 5 hr with 2.5 g adsorbent dosage.

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