ADSORPTION STUDIES OF Fe[III] ON ACTIVATED CHARCOAL IN THE PRESENCE OF LACTIC ACID AS CHELATING AGENT

Dissertation submitted in Partial fulfillment for the requirements for the award of degree of

Bachelor of Science
in
CHEMISTRY

By

1. **MukthiKanth Rout, B.Sc(Chemistry), III Year**
2. **S.Raja Ramesh, B.Sc(Chemistry), III Year**
3. **P.Sujil Kumar, B.Sc(Chemistry), III Year**
4. **Ch.srinivas, B.Sc(Chemistry), III Year**
5. **B.Mahesh Kumar, B.Sc(Chemistry), III Year**
6. **B.NikhileshBabu, B.Sc(Chemistry), III Year**

Under the Guidance:

Dr. K.ABHIJIT
HEAD
DEPARTMENT OF CHEMISTRY

TARA GOVERNMENT COLLEGE, SANGAREDDY (A)
2021-22

CERTIFICATE

This is to certify that the project work entitled "**ADSORPTION STUDIES OF Fe[III] ON ACTIVATED CHARCOAL IN THE PRESENCE OF LACTIC ACID AS CHELATING AGENT** " is presented by B.Sc (CHEMISTRY) students in partial fulfillment of the requirements for the degree of Bachelor of Science in Chemistry by the Tara Govt. College, Sangareddy(A) (Affiliated to Osmania University, Hyderabad) during the academic year 2021-2022.

The results embodied in this report have not been to any other University or Institution for the award of any degree.

(Dr. K. ABHIJIT)

Project Supervisor& Head, Department of Chemistry
Tara Government College, Sangareddy(A)

EXTERNAL EVALUATOR

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1. **MukthiKanth Rout, B.Sc(Chemistry), III Year**
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5. **B.Mahesh Kumar, B.Sc(Chemistry), III Year**
6. **B.NikhileshBabu, B.Sc(Chemistry), III Year**

DECLARATION

We hereby declare that the project report entitled "ADSORPTION STUDIES OF Fe[III] ON ACTIVATED CHARCOAL IN THE PRESENCE OF LACTIC ACID AS CHELATING AGENT" is the work done by us in the campus at ***Department of Chemistry****, Tara Government College, Sangareddy(A) during the academic year 2021-2022 and is submitted in partial fulfillment of the requirements for the degree of* ***Bachelor of Science*** *by Tara Govt. College, Sangareddy(A) (Affiliated to Osmania University, Hyderabad) during the academic year 2021-2022.*

S.No.	Name of the Student	Roll Number	Group	Year	Signature
1	MukthiKanth Rout	6058-19-578-022	B.sc - MCCS	III	
2	S. Raja Ramesh	6058-19-578-030	B.sc - MCCS	III	
3	P. Sujil Kumar	6058-19-578-027	B.sc - MCCS	III	
4	Ch. Srinivas	6058-19-578-008	B.sc - MCCS	III	
5	B. Mahesh Kumar	6058-19-578-004	B.sc - MCCS	III	
6	B. NikhileshBabu	6058-19-578-005	B.sc - MCCS	III	

TARA GOVERNMENT COLLEGE, SANGAREDDY – 502 001
(AUTONOMOUS)

BONAFIDE CERTIFICATE

Certified that the project report"ADSORPTION STUDIES OF Fe[III] ON ACTIVATED CHARCOAL IN THE PRESENCE OF LACTIC ACID AS CHELATING AGENT" is the bonafide work of

1. **MukthiKanth Rout, B.Sc(Chemistry), III Year**
2. **S.Raja Ramesh, B.Sc(Chemistry), III Year**
3. **P.Sujil Kumar, B.Sc(Chemistry), III Year**
4. **Ch.srinivas, B.Sc(Chemistry), III Year**
5. **B.Mahesh Kumar, B.Sc(Chemistry), III Year**
6. **B.NikhileshBabu, B.Sc(Chemistry), III Year**

who carried out the project work under my supervision.

Dr.K.ABHIJIT	*Smt. M.PRAVEENA*
PROJECT SUPERVISOR	***PRINCIPAL***

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Chapter-I

INTRODUCTION

Adsorption

Adsorption operation involves the touch of solids with both liquids and gases in which the mass switch is towards solids. The reverse of this operation is known as "desorption." Adsorption operations exploit the potential of positive solids to concentrate precise materials from fluid on to their surfaces. The adsorbed substance is known as an adsorbate, and the solid substance is known as an adsorbent. Typical packages of this stable-liquid operation are as follows:

- Gas: Elimination of moisture dissolved in gas
- de-colorization of petroleum merchandize and sugar answers
- water for the removal of objectionable flavours and odours.

The stable-gasoline operations consist of:

- Dehumidification of air and gases
- Gas purification is the process of removing noxious odours and impurities from gases.
- The recovery of precious solvent vapours from dilute gasoline combinations
- fractionate combinations of hydrocarbon gases, inclusive of methane, ethane, and propane.

NATURE OF ADSORBENTS

Adsorbents are commonly in granular form, with their size starting from zero to five to twelve millimetres. They must neither provide a high strain drop nor get overly excited by the flowing stream. They should now not lose their shape and size at the same time as managing. They must have a large surface area per unit mass and a lot of pores.

S.NO	Adsorbent	Source	Application
1.	Fuller's earth	Naturally occurring clay is heated and dried to get a porous structure.	De-colorizing, drying of lubricating oils, kerosene and engine oils.
2.	Activated clay	Bentonite or other activated clay which are activated by treatment with sulfuric acid and further washing, drying and crushing..	Used for de-colorizing petroleum products.
3.	Bauxite	A naturally occurring hydrated alumina, activated by heating at 230-815^{0}C	Used for de-colorizing petroleum products and for

			drying gases.
4.	Alumina	Hard hydrated aluminum oxide is activated by heating to drive off the moisture and then crushed to the desired size.	Used as a desiccant.
5.	Bone – Char	Obtained by destructive distillation of crushed bones at 600-9000C.	Used for refining sugar and can be reused after washing and burning.
6.	Activated Carbon	(i) Vegetable matter is mixed with calcium chloride, carbonized and finally, the inorganic compounds are leached away. (ii) Organic matter is mixed with	De-colorizing of sugar solutions, chemicals, drugs, water purification, refining of vegetable and animal oils, recovery of gold and silver from cyanide ore-leach

		porous pumice stones and then heated and carbonized to deposit the carbonaceous matter throughout the porous particle. (iii) Carbonizing substances like wood, sawdust, coconut shells, fruits pits, lignite, and subsequent activation with the hot air steam. it is available in granular or pelleted form.	solution, recovery of solvent vapour from gas-mixtures, collection of gasoline hydro-carbons from natural gas, fractionation of hydrocarbon gases.
7.	Silica gel	A hard granular and porous product obtained from sodium silicate solution after treatment with acid.	Used for de-hydration of air and other gases, fractionation of

		Normally has 4 to 7% water in the product.	hydrocarbons.
8.	Molecular sieves	These are porous synthetic zeolite crystals, metal aluminosilicates.	Dehydration of gases and liquids, and separation of gas— liquid hydrocarbon mixture.

Heat of adsorption

The differential heat of adsorption (—H) is described as the heat liberated at a regular temperature whilst a unit quantity of vapour is adsorbed on a large quantity of stable already containing adsorbate. The solid so used is in such a huge quantity that the adsorbate concentration stays unchanged.

The indispensable warmness of adsorption (AH) at any concentration X is described as the enthalpy of the adsorbate—adsorbent mixture minus the sum of the enthalpies of unit weight of natural stable adsorbent and sufficient pure adsorbed substance (before adsorption) to offer the specified concentration X at an equal temperature.

The differential warmness of adsorption and the necessary heat of adsorption are capabilities of temperature and adsorbate concentration.

Effect of temperature

An increase in temperature at steady stress decreases the quantity of solute adsorbed from a combination. However, the generalisation of the end result isn't smooth.

Effect of pressure

Generally, reducing stress reduces the quantity of adsorbate adsorbed upon the adsorbent. However, the relative adsorption of paraffin hydrocarbons on carbon decreases at high pressures.

Liquids

The pollutants are available both at low and high fixations in fluids. These are typically eliminated by an adsorption procedure. The attributes of adsorption of low and high fixation debasements are unique.

Adsorption of solute from Dilute solutions

Whenever a combination of solute and dissolvable is adsorbed, utilising an adsorbent, both the dissolvable and the solute are adsorbed. Because of this, the main family member or evident adsorption of solute cannot be entirely set in stone.

Thus, it is a typical practice to treat a known volume of an arrangement of unique focus (Co) with a known load of adsorbent. Allow C* to be the last harmony grouping of solfege in the arrangement.

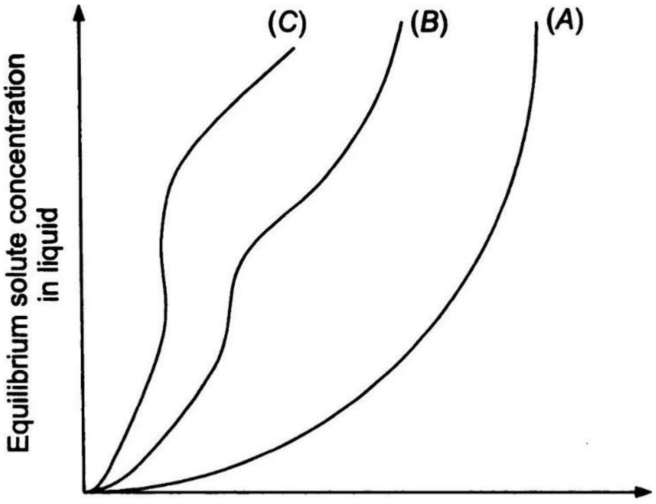
Assuming that v is the volume of arrangement per unit mass of adsorbent (cc/g) and Co and C* are the underlying and balance focuses (g/cc) of the solute, then the evident adsorption of solute per unit mass of adsorbent, dismissing any adjustment of volume, is v (Co-C*), (g/g).

This articulation is, for the most part, pertinent to weakening arrangements. At the point when the negligible part of the first dissolvable which can be adsorbed is small, the C* esteem relies upon the temperature, nature, and properties of the adsorbent.

On account of weakening arrangements and over a small focus range, the Freundlich adsorption isotherm portrays the adsorption peculiarities.

The Freundlich adsorption condition is additionally very helpful in situations where the real character of the solute isn't known, for example, the expulsion of shading substances from sugar arrangements, oils, and so on. The arrangement's variety content can be easily estimated by utilising a spectrophotometer or colorimeter. The translation of this information is shown in working model 2. Assuming the value of n is high, i.e., 2 to 10, adsorption is great. Assuming it lies between I and 2, tolerably troublesome and short of what I demonstrate, I demonstrate unfortunate adsorption attributes. A normal adsorption isothermal for the adsorption of different adsorbates A, B, and C in a weakened arrangement at a similar temperature for a similar adsorbent is displayed in Fig.

(A)



Kg solute apparently adsorbed / kg adsorbent

Fig. Adsorption isotherms for various adsorbents.

Significance of Research problem

Adsorption of heavy metals is a key development approach for more advanced remediation technologies for the preservation of the environment. However, the effectiveness of heavy metal adsorption under typical conditions using a good adsorbent depends on a number of variables that must be precisely adjusted to get an effective adsorption process. The adsorption of heavy metals was boosted in the presence of suitable facilitating agents, which undoubtedly improved the heavy metal procedures already in use.

Objective of the Project

To evaluate the impact of Lactic acid as chelating agent in the adsorption of Fe (III) from aqueous solution by activated charcoal as an adsorbent to develop efficient remediation technology using Metal-ligand interactions.

Chapter-II

ADSORPTION OF HEAVY METALS USING ACTIVATED CHARCOAL

Nowadays, various heavy metals have been released into the waters, causing serious pollution of water resources and endangering human health. Therefore, it is very important to study the removal of heavy metals from water. In this work, the adsorption of heavy metal ions including Pb(II), Cu(II), Zn(II), Cd(II) and Cr(VI) onto activated carbon (AC) from aqueous solutions was investigated in both single and mixed systems. The batch adsorption experiment for heavy metal ions on AC in single system was studied at different conditions including contact time, initial concentration, initial pH and adsorbent dosage. The results indicated that the adsorption kinetics and isotherms followed the pseudo-first order model and the Langmuir isotherm model, respectively. The final removal efficiencies and maximum adsorption capacity followed the order of Pb(II)> Cr(VI) Cd(II) > Cu(II) > Zn(II) at the pH 5.0 and 25°C in single system. The adsorption of heavy metals was mainly determined by the pH of the solution and the surface properties of the adsorbent, and it was found that the initial pH of 3 was optimal for the removal of Cr(VI), which was different from the optimum initial pH of 7.0 for Pb(II), Cu(II), Zn(II) and Cd(II). The results demonstrated that the electrostatic interaction between the surface of AC and heavy metal ions played an important role in the adsorption of heavy metal ions. In addition, compared with the results in single system, the results in multi-component mixed system showed that the adsorption of Cu(II) and Cr(VI) was promoted, but the adsorption of Zn(II) and Cd(II) was inhibited. The factors affecting the adsorption impetuses are interactive involving electrostatic interaction, promotion or inhibition between heavy metal ions, chemical reaction and so on.

Adsorption of Iron complex using Activated Charcoal

Among pollutants, iron have attracted our attention because; they are the most released by industries. To curb this kind of pollution, a local alternative must be developed by these countries to ensure the health of their inhabitants.

There are several methods of treatment geared towards removing these pollutants from water, such as: filtration, ozonation, clarification, photocatalysis, adsorption, membrane processes, electro coagulation, and chemical processes. Although, the above-mentioned methods are efficient in treating high concentration of heavy metal ions, nevertheless these techniques also have disadvantages including incomplete metal removal, high consumption of reagent and very high cost incurred in the process. For the lower concentration of heavy metal ions, adsorption is a much preferable technique and activated carbon has been widely applied for treating industrial wastewater.

Activated carbons as adsorbents from plant origin have been developed from agricultural by-products, called biosorbent. Crude olive stone was used for adsorption of iron present in industrial wastewater. Adsorption on activated carbon remains one of the mainly used methods for these countries.

The aim of this work is to investigate the removal of iron from synthetic solution and industrial wastewater with commercial activated carbon from local wood. The adsorption of metal showed two important parameters: initial concentration and pH of solution. These parameters allows for maximizing the amount of adsorbed metals. However, the problems associated with these adsorbents are the regeneration and recovery of the useful materials, which makes them unattractive for wider commercial applications.

The carboxyl group as a part of many compounds (pharmaceuticals, supplements) is a "universal" O-donor ligand which can form complexes of different coordination, and can act as a monodentate or a bidentate ligand.

α-Hydroxycarboxylic acids are constitutive components of many biochemical processes in the living world. They are often used in pharmaceutical preparations. Lactic acid, 2hydroxypropanoic acid, (LA) is formed by fermentation of sugars and other carbohydrates under the action of anaerobic bacteria and it can be ingested with food into the body. In medicine it is used as a component of the Ringer's and Hartman's solutions. Lactic acid is a suitable molecule for model systems for the study of interaction of biometals with O-donor ligands in biosystems. With M(II) metal ions from solutions of molar concentrations, depending on the conditions of complexation, properties and synergistic effect of the metal ions, LA can form different types of complex associates With M(II) dmetal ions LA can form complexes of the type $[M(LA)_2R]$, where it behaves as a bidentate ligand, and R is molecule of solvent . Cu(II) ion can form mononuclear and binuclear complexes with aliphatic α-hydroxycarboxylic acids at pH values .

Previous studies have shown that there is a mobility of metal ions from biological hard mineral tissue of teeth that had been exposed to the impact of various media (lactic acid, acetic acid, etc.) as a result of the interaction of metal ions with the test media . In the literature there are no enough data about interactions of d-metals and LA under approximately physiological conditions, and at the level of micro-molar concentration at which they are present in human body.

Iron - Lactic acid complex in aqueous solutions

The photochemistry of a I: I Fe [lll]—lactic acid complex $\mathrm{Fe(Lact)]^{+}}$, in aqueous solutions was studied by stationary photolysis, nanosecond laser flash photolysis (355 nm, 6 ns), and femtosecond pump—probe spectroscopy (400 nm, 200 fs). The quantum yield of photolysis of $\mathrm{Fe(Lact)]^{+}}$, upon excitation at 355 nm is 0.4 and 0.22 in the deoxygenated and air-saturated solutions, respectively. Weak transient absorption in the range of 500—750 nm was observed in the nanosecond experiments. It was assigned to a $\mathrm{Fe^{II}\ldots{}^{-}O{-}CH(Me){-}COO^{\cdot}]^{+}}$ radical complex. The spectral properties of the ligand-to-metal charge transfer excited state and the characteristic time of formation of the radical complex (1.5 ps) were determined in the femtosecond spectroscopy experiments. A reaction mechanism was proposed, which involves innersphere electron transfer in the excited complex with the formation of a radical complex $\mathrm{Fe^{II}\ldots{}^{-}O{-}CH(Me){-}COO^{\cdot}]^{+}}$ and its subsequent transformation to the end product of the photochemical reaction.

The photochemistry of Fe [lll] carboxylate complexes with natural carboxylic acids (tartaric, pyruvic, lactic, succinic, as well as humic and fulvic acids) is of considerable interest from the standpoint of environmental photochemistry and photocatalysis because photolysis of these compounds is accompanied by the formation of reactive $\mathrm{H_2O_2)^{1-5}}$ oxygen species (•OH, H02•), which leads to mineralization of organic matter and to CO and C02 generation in natural water.

A traditional mechanism of photolysis of Fe [lll] carboxylate complexes implies that the primary photochemical event involves inner-sphere electron transfer accompanied by reduction of Fe [lll] to Fe [ll] and escape of a free radical into the solvent bulk, followed by fast decarboxylation of the radical. A secondary radical generated as a result of decarboxylation reacts with Fe [lll] complexes and dissolved oxygen to give reactive oxygen species.

This mechanism is based on the analysis of photochemical reaction end products, and the generation of organic radicals in the primary photochemical process was frequently not confirmed experimentally. Simultaneously, studies on the photochemistry of iron(ll) complexes with oxalic acid 13 and ethylene diamine tetra acetic acid 12'14 using pulsed methods enabled the formulation of an alternative photolysis mechanism based on the production of a long-lived radical complex [Fe^{ll}s • OOC—R] ^{2}+ in the primary photochemical event.

In this work, we studied primary photochemical processes, taking a Fe^{lli}complex with lactic acid as an example. The emphasis is placed on the detection of corresponding radical complex and its precursor, i.e., the excited state, and on the determination of their spectral and kinetic properties.

Chapter-III

ATOMIC ABSORPTION SPECTROMETRY

Atomic absorption spectrometry (AAS) is an analytical technique that measures the concentrations of elements. Atomic absorption is so sensitive that it can measure down to parts per billion of a gram (µg dm^{-3}) in a sample. The technique makes use of the wavelengths of light specifically absorbed by an element. They correspond to the energies needed to promote electrons from one energy level to another, higher, energy level.

Atomic absorption spectrometry has many uses in different areas of chemistry.

Clinical analysis, analysing metals in biological fluids such as blood and urine. **Environmental analysis** monitoring our environment –*eg* finding out the levels of various elements in rivers, seawater, drinking water, air, petrol and drinks such as wine, beer and fruit drinks.

Pharmaceuticals. In some pharmaceutical manufacturing processes, minute quantities of a catalyst used in the process (usually a metal) are sometimes present in the final product. By using AAS the amount of catalyst present can be determined.

Industry. Many raw materials are examined and AAS is widely used to check that the major elements are present and that toxic impurities are lower than specified – *eg* in concrete, where calcium is a major constituent, the lead level should be low because it is toxic.

Mining. By using AAS the amount of metals such as gold in rocks can be determined to see whether it is worth mining the rocks to extract the gold.

How it works

Atoms of different elements absorb characteristic wavelengths of light. Analysing a sample to see if it contains a particular element means using light from that element. For example with lead, a lamp containing lead emits light from excited lead atoms that produce the right mix of wavelengths to be absorbed by any lead atoms from the sample. In AAS, the sample is atomised –*ie* converted into ground state free atoms in the vapour state – and a beam of electromagnetic radiation emitted from excited lead atoms is passed through the vaporised sample. Some of the radiation is absorbed by the lead atoms in the sample. The greater the number of atoms there is in the vapour, the more radiation is absorbed. The amount of light absorbed is proportional to the number of lead atoms. A calibration curve is constructed by running several samples of known lead concentration under the same conditions as the unknown. The amount the standard absorbs is compared with the calibration curve and this enables the calculation of the lead concentration in the unknown sample.

Consequently an atomic absorption spectrometer needs the following three components: a light source; a sample cell to produce gaseous atoms; and a means of measuring the specific light absorbed.

The light source

The common source of light is a ‘hollow cathode lamp’ *(Fig. 1*).

Figure1

This contains a tungsten anode and a cylindrical hollow cathode made of the element to be determined. These are sealed in a glass tube filled with an inert gas –*eg* neon or argon – at a pressure of between 1 Nm^{-2} and 5 Nm^{-2}. The ionisation of some gas atoms occurs by applying a potential difference of about 300–400 V between the anode and the cathode. These gaseous ions bombard the cathode and eject metal atoms from the cathode in a process called sputtering. Some sputtered atoms are in excited states and emit radiation characteristic of the metal as they fall back to the ground state – *eg*$Pb^* \rightarrow Pb + h\nu$ (*Fig. 2*).

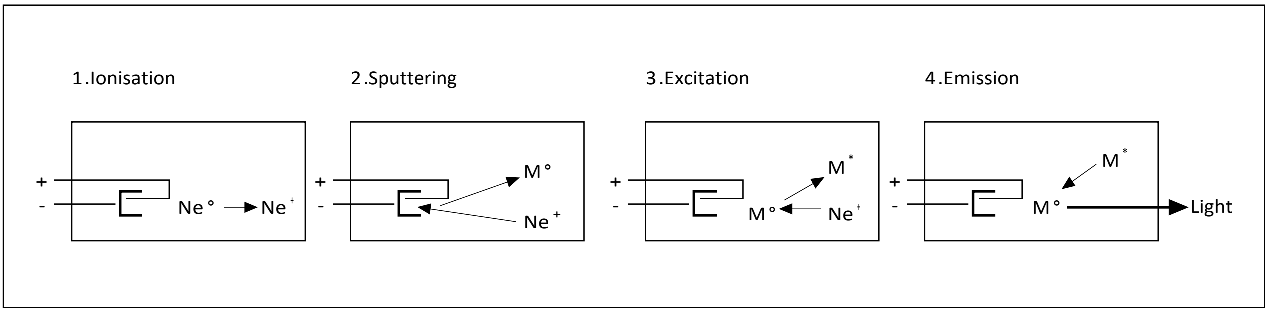


Figure. 2

The shape of the cathode concentrates the radiation into a beam which passes through a quartz window, and the shape of the lamp is such that most of the sputtered atoms are redeposited on the cathode.

A typical atomic absorption instrument holds several lamps each for a different element. The lamps are housed in a rotating turret so that the correct lamp can be quickly selected.

The optical system and detectorA mono chromator is used to select the specific wavelength of light – ie spectral line – which is absorbed by the sample, and to exclude other wavelengths. The selection of the specific light allows the determination of the selected element in the presence of others. The light selected by the monochromator is directed onto a detector that is typically a photomultiplier tube. This produces an electrical signal proportional to the light intensity (Fig. 3)

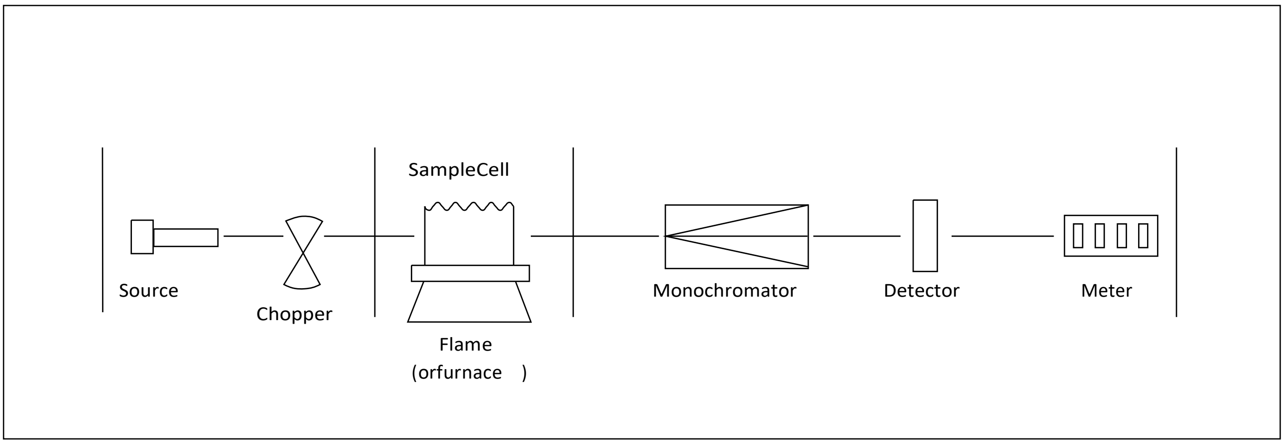


Figure 3

Double beam spectrometers

Modern spectrometers incorporate a beam splitter so that one part of the beam passes through the sample cell and the other is the reference *(Fig. 4)*. The intensity of the light source may not stay constant during an analysis. If only a single beam is used to pass through the atom cell, a blank reading containing no analyte (substance to be analysed) would have to be taken first, setting the absorbance at zero. If the intensity of the source changes by the time the sample is put in place, the measurement will be inaccurate. In the double beam instrument there is a constant monitoring between the reference beam and the light source. To ensure that the spectrum does not suffer from loss of sensitivity, the beam splitter is designed so that as high a proportion as possible of the energy of the lamp beam passes through the sample.

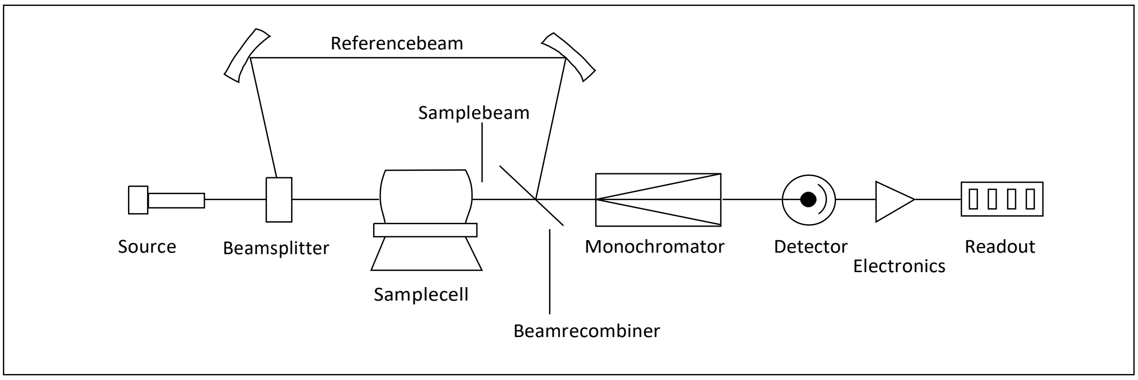


Figure 4

Atomisation of the sample Two systems are commonly used to produce atoms from the sample. Aspiration involves sucking a solution of the sample into a flame; and electrothermal atomisation is where a drop of sample is placed into a graphite tube that is then heated electrically.

Some instruments have both atomisation systems but share one set of lamps. Once the appropriate lamp has been selected, it is pointed towards one or other atomisation system.

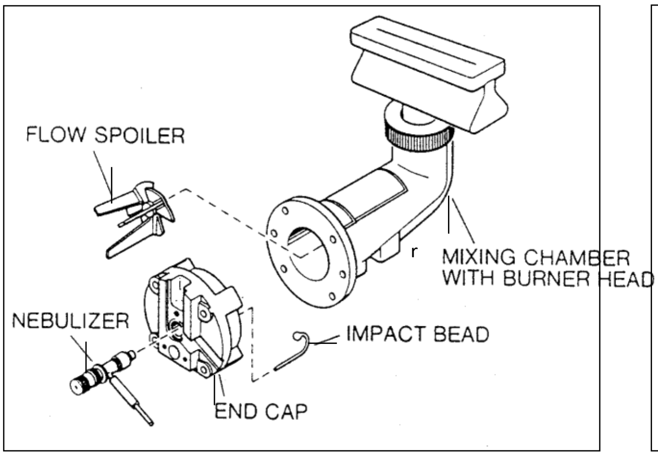


Figure5

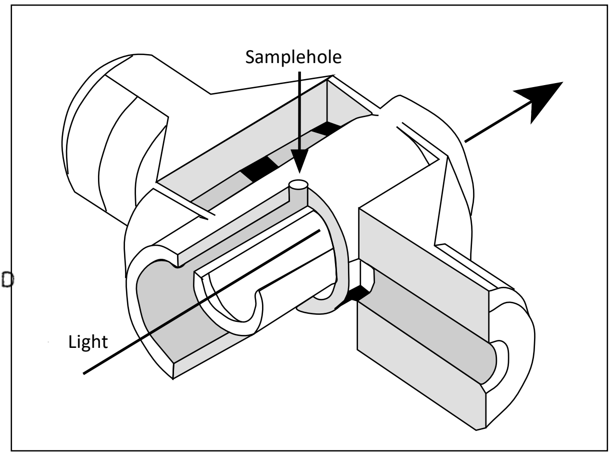


Figure6

Flame aspiration

Figure 5 shows a typical burner and spray chamber. Ethyne/air (giving a flame with a temperature of 2200–2400°C) or ethyne/dinitrogen oxide (2600– 2800°C) is often used. A flexible capillary tube connects the solution to the nebuliser. At the tip of the capillary, the solution is 'nebulised' –*i.e.* broken into small drops. The larger drops fall out and drain off while smaller ones vaporise in the flame. Only *ca* 1% of the sample is nebulised.

Electro thermal atomisation of the original sample*Figure 7* shows a flame atomic *Figure 6* shows a hollow graphite tube with a platform. Absorption spectrometer with an auto sampler and 25 µl of sample (*ca* 1/100th of a raindrop) is placed through the sample hole and onto the platform from an automated micropipette and sample changer. The tube is heated electrically by passing a current through it in a pre-programmed series of steps. The details will vary with the sample but typically they might be 30–40 seconds at 150°C to evaporate the solvent, 30 seconds at 600°C to drive off any volatile organic material and char the sample to ash, and with a very fast heating rate (*ca* 1500 °C s^{-1}) to 2000– 2500°C for 5–10 seconds to vaporise and atomise elements (including the element being analysed). Finally heating the tube to a still higher temperature –*ca* 2700°C – cleans it ready for the next sample. During this heating cycle the graphite tube is flushed with argon gas to prevent the tube burning away. In electro thermal atomisation almost 100% of the sample is atomised. This makes the technique much more sensitive than flame AAS.

Sample preparation

Sample preparation is often simple, and the chemical form of the element is usually unimportant. This is because atomisation converts the sample into free atoms irrespective of its initial state. The sample is weighed and made into a solution by suitable dilution. Elements in biological fluids such as urine and blood are often measured simply after a dilution flow injection accessory.

When making reference solutions of the element under analysis, for calibration, the chemical environment of the sample should be matched as closely as possible – *i.e.* the analyte should be in the same compound and the same solvent. Teflon containers may be used when analysing very dilute solutions because elements such as lead are sometimes leached out of glass vessels and can affect the results

Background absorption

It is possible that other atoms or molecules apart from those of the element being determined will absorb or scatter some radiation from the light source. These species could include un vaporised solvent droplets, or compounds of the matrix (chemical species, such as anions, that tend to accompany the metals being analysed) that are not removed completely. This means that there is background absorption as well as that of the sample.

One way of measuring and correcting this background absorption is to use two light sources, one of which is the hollow cathode lamp appropriate to the element being measured. The second light source is a deuterium lamp.

The deuterium lamp produces broad band radiation, not specific spectral lines as with a hollow cathode lamp. By alternating the measurements of the two light sources – generally at 50 –100 Hz – the total absorption (absorption due to analyte atoms plus background) is measured with the specific light from the hollow cathode lamp and the background absorption is measured with the light from the deuterium lamp. Subtracting the background from the total absorption gives the absorption arising from only analyte atoms.

Figure 7

Calibration

A calibration curve is used to determine the unknown concentration of an element –*eg* lead – in a solution.

The instrument is calibrated using several solutions of known concentrations. A calibration curve is produced which is continually rescaled as more concentrated solutions are used – the more concentrated solutions absorb more radiation up to a certain absorbance. The calibration curve shows the concentration against the amount of radiation absorbed *(Fig. 8(a))*.

The sample solution is fed into the instrument and the unknown concentration of the element – *e.g.* lead – is then displayed on the calibration curve *(Fig. 8(b))*.

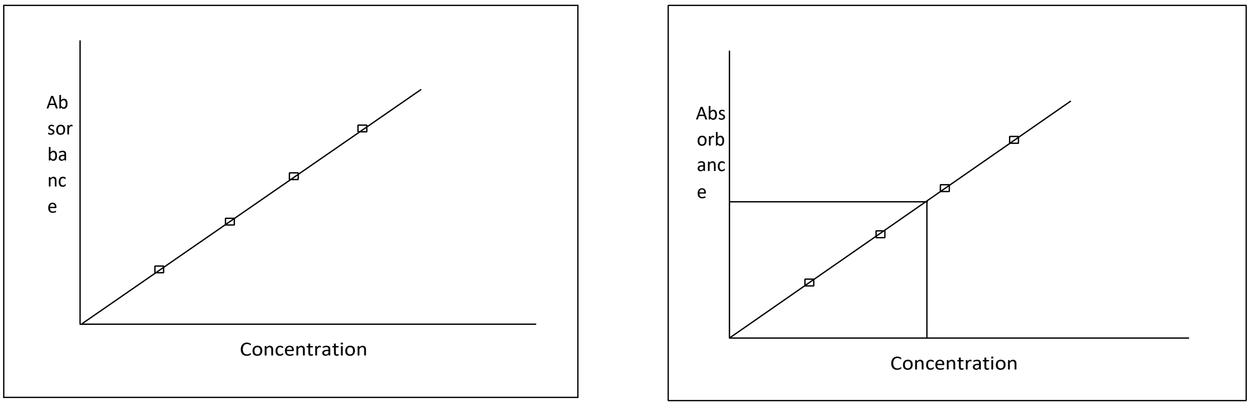


Figure 8(a) and Figure 8(b)

Interferences and matrix modification other chemicals that are present in the sample may affect the atomisation process. For example, in flame atomic absorption, phosphate ions may react with calcium ions to form calcium pyrophosphate. This does not dissociate in the flame and therefore results in a low reading for calcium. This problem is avoided by adding different reagents to the sample that may react with the phosphate to give a more volatile compound that is dissociated easily. Lanthanum nitrate solution is added to samples containing calcium to tie up the phosphate and to allow the calcium to be atomised, making the calcium absorbance independent of the amount of phosphate. With electrothermal atomisation, chemical modifiers can be added which react with an interfering substance in the sample to make it more volatile than the analyte compound. This volatile component vaporises at a relatively low temperature and is removed during the low and medium temperature stages of electrothermal atomisation.

Chapter-IV

METHODOLOGY

Required materials:

- $FeCl_3.6H_2O$ (MOLYCHEM)
- Lactic acid (AVRA)
- Activated charcoal (SDFCL)
- Ultra-pure water (demineralized)
- Distilled water
- 2% nitric acid
- Volumetric flask – 250 mL (Borosilicate glass)
- Watch glass
- Hot air oven (BIO-TECHNICS INDIA)
- Digital weighing machine (CITIZEN)
- AAS (ICE 3300)

Procedure:

- Using a 250ml volumetric flask, prepare 100 ppm of Ferric chloride hexahydrate metal solution.
- For the preparation of 100 ppm of Ferric chloride hexahydrate metal solution we have to calculate the amount of Ferric chloride hexahydrate required for the solution.
- Now add 3 drops of lactic acid in the Ferric chloride hexahydrate metal solution using a dropper, then we obtain the metal ligand solution which will be in 1:2 ratio.
- Keep the solution for 24 hrs. for better results.
- Now take 100ml of metal ligand solution in a 500ml beaker and add 5 grams of Activated Charcoal to it.
- Keep this solution aside for 48 hours. To obtain a metal ligand solution absorbed charcoal, filter the solution using a filter paper and rinse it 3 times using distilled water.
- Take the metals adsorbed activated Charcoal on a watch glass and keep it in the oven

at 60ºC for 10 hours to get rid of moisture.

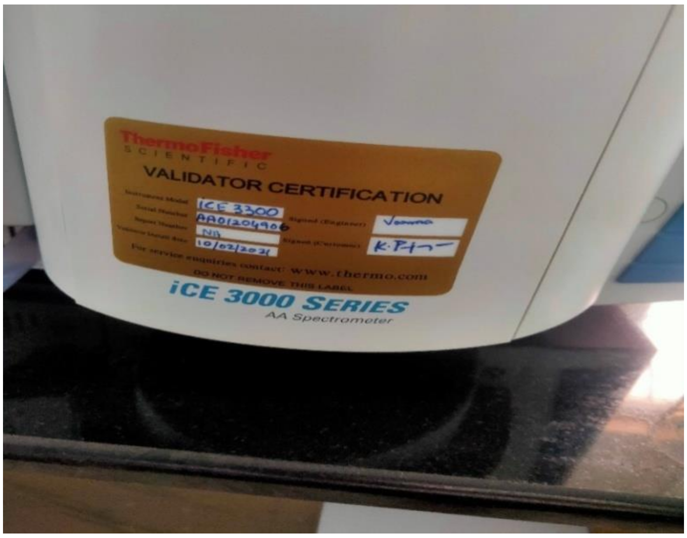
- After getting rid of moisture, we get fine powder of metal adsorbed activated Charcoal.
- Later shift the compound to a glass valve.
- Now send the adsorbent sample to the AAS for further tests.
- Here in AAS an Atomic Absorption Spectrometer is used for the tests, which consists of different bulbs that have wavelengths of different elements.
- The spectrometer cannot intake the solid particles, it can only intake the liquid.
- So the given adsorbent sample is mixed with the nitric acid solution.
- Now the adsorbent sample that is mixed with nitric acid is heated in the fume hood at 100°C for 15 minutes.
- The test sample is reduced to slurry liquid.
- Not all of the adsorbent samples given at the AAS are mixed, but 0.5 gm. of the adsorbent sample is diluted in 25mL of nitric acid solution (in which the nitric acid is present 2% in the ultra-pure water).
- Now filter the slurry liquid in thick filter paper. The ultra-pure water is used in rinsing of the adsorbent sample to 25mL, as the sample that should be used in the spectrometer has reduced because of the heating.
- Rinsing the slurry liquid with the ultra-pure water also helps the metal ions present in the adsorbent sample, flow with the nitric acid present in the water for the test purpose.
- Now 5 test standards are prepared using 1ppm, 2ppm, 3ppm, 4ppm and 5ppm “Fe” in it.
- Firstly the spectrometer is checked using only ultra-pure water with 2% nitric acid

present in it.

- After that it is tested with the 5 different prepared Fe standards.
- Now our test sample is tested after the 5 Fe standards are tested.
- The bulb present in the spectrometer is set at the same wavelengths as of the “Fe”.
- When the test sample is being tested the bulb that consists of the “Fe” wavelength automatically transmits the same wavelengths through the sample.
- When the test sample is being tested in the spectrometer the Fe ions present with nitric acid are burned with the burner present in the spectrometer.
- As the concentration of the Fe increases the temperature also increases and the color gradually turns from orange to red and red to crimson and so on… The average temperature is around 1500°C.
- The waste present in the test sample other than Fe and nitric acid after the completion of tests are collected in the waste collector present below.
- The signal from the spectrometer can be seen in the monitor, which is already connected to the spectrometer.
- The signal from the spectrometer can be recorded and calculated from the graphs formed and numeric values shown in the monitor.
- Same Experiment carried out without the interference/addition of Ligand i.e. Lactic acid for Control Experiment.

<u>AA spectrometer</u> (a)

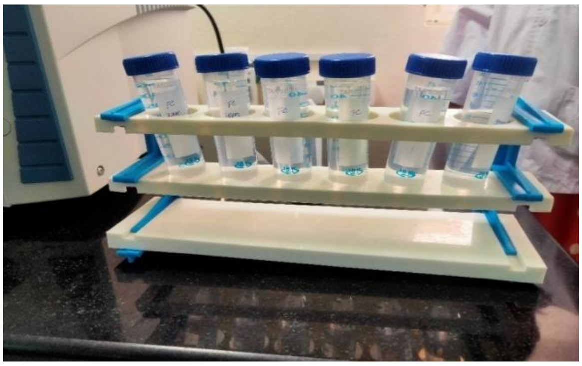
<u>AA spectrometer</u> (b)



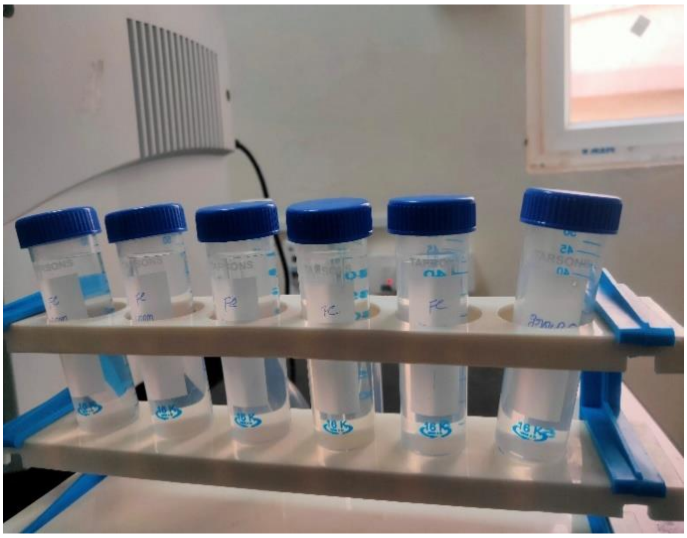
Model no. ICE 3300

Fumehood (a)

Fume hood (b)



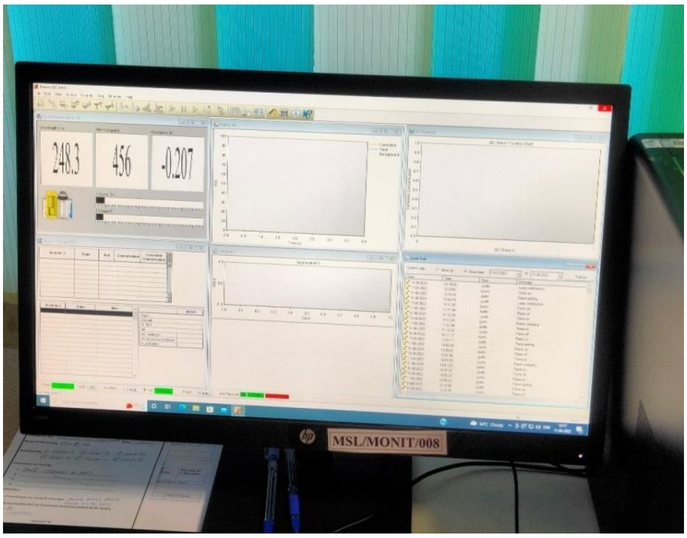
Fe standards with test sample



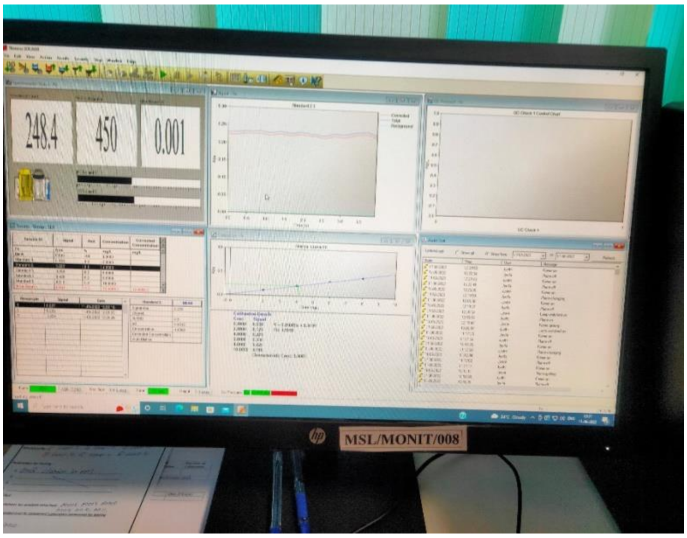
Fe standards with test sample

Burning of Nitric acid solution in AA Spectometer

Burning of Test sample in AA Spectometer

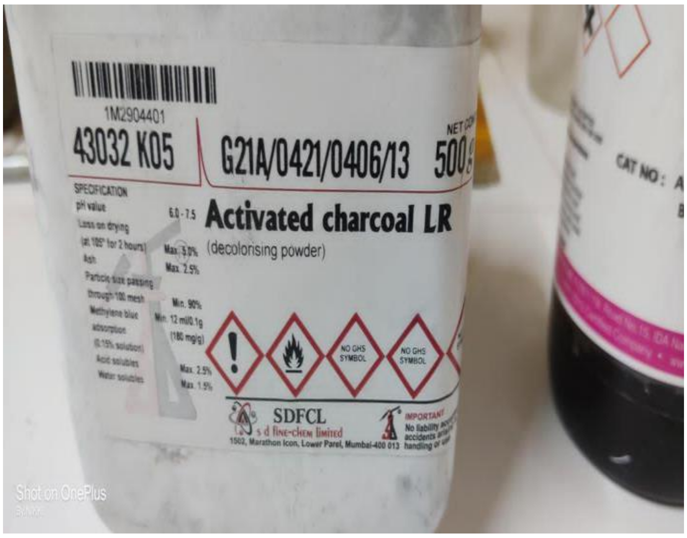


Values before intake of test samples



Values after intake of test samples

Lactic acid



Activated charcoal

Metal ligand solution (a)

Glass plate

Spatula

Measuring Tube

Hot air Oven

5gm of Activated charcoal in metal ligand solution

Metal adsorbed charcoal

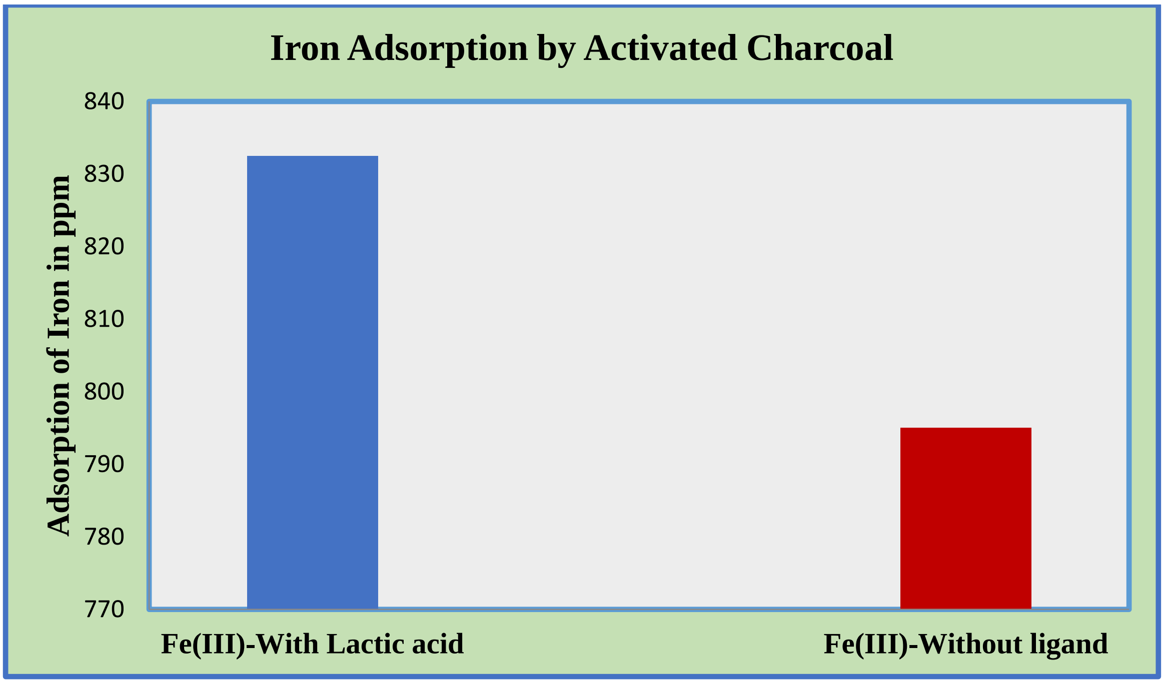
Test Samples


MNR

Chapter-V

RESULTS AND DISCUSSION

Activated charcoal adsorbed **832.5** ppm of Iron metal from aqueous solution of Fe(III)-Lactic acid metal ligand solution. Whereas, Activated charcoal adsorbed only **795.0** ppm when Lactic acid is absent. It is evident from the AAS results, ligand involvement enhanced the metal adsorption by initiating potential chemical interactions between adsorbate and adsorbent. Lactic acid strongly form coordination bonding with Fe(III) to form a soluble complex in aqueous solution. The ligand capped Fe(III) complex strongly adsorbed to Activated charcoal by establishing chemical bonding. From the AAS results, it is conclusive that **4.71** % of adsorption increased in the presence of Lactic acid as chelating agent.



Impact of Lactic acid on Adsorption of Fe (III) ions from aqueous solution by Activated Charcoal

Spectrometer Parameters – Fe:

Element : Fe	Measurement mode : Absorbance	
Wavelength : 248.3nm	Band pass : 0.2nm	Lamp current : 75%
Background correction : D2	High Resolution : Off	Optimise Spectrometer Parameters : No
Signal type : continuous	Resamples : Fast	Number of resamples : 3
Measurement time : 4.0secs	Flier mode : No	
Use RSD Test : No		

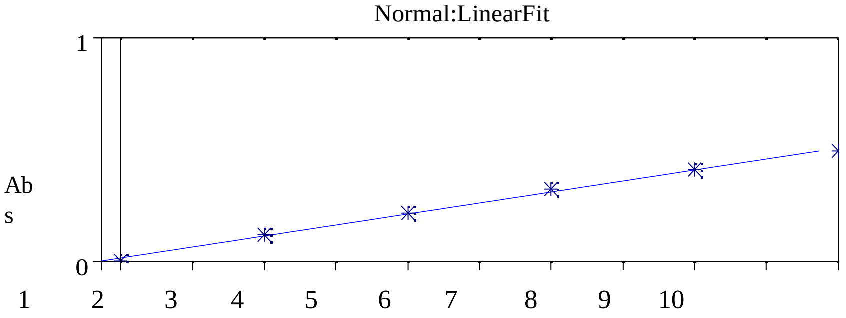
Flame Parameters – Fe:

Flame type : Air – C2H2	Fuel Flow : 0.9L/min	Auxiliary Oxidant : Off
Nebuliser Uptake : 4secs	Bunsen Stabilisation : 0mins	Optimise Fuel Flow : No
Burner Height :7.0mm	Optimise Burner fuel : No	

Calibration Parameters–Fe:

Calibration mode : Normal	Line fit : Linear	Use stored calibration : No
Concentration units : mg/L	Scales units : mg/L	Scaling factor : 1.0000
Acceptable fit : 0.990	Rescale Limits : 10.0%	Failure Action : Flag and Continue
Standard 1 - 2.0000	Standard 4 - 8.0000	
Standard 2 - 4.0000	Standard 5 - 10.0000	
Standard 3 - 6.0000		

Solutions Results–Fe:



Y = 0.04924x + 0.0141
Fit: 0.9965
Characteristic Conc.: 0.0894

Sample ID	**Signal**	**RSD**	**Conc.**
	Abs	**%**	**Mg/L**
Fe Blank	0.001	35.6	0.0000
1	0.001	Background: -0.003	
2	0.001	Background: -0.003	
3	0.000	Background: -0.003	
Fe Standard 1	0.120	0.3	2.0000
1	0.120	Background: 0.003	
2	0.119	Background: 0.003	
3	0.120	Background: 0.003	
Fe Standard 2	0.215	0.3	4.0000
1	0.215	Background: 0.004	
2	0.215	Background:0.004	
3	0.216	Background:0.004	
Fe Standard 3	0.322	0.2	6.0000
1	0.323	Background: 0.005	
2	0.322	Background: 0.005	
3	0.322	Background: 0.005	
Fe Standard 4	0.411	0.3	8.0000
1	0.410	Background: 0.005	
2	0.412	Background: 0.005	
3	0.412	Background: 0.005	
Fe Standard 5	0.493	0.1	10.0000
1	0.494	Background: 0.005	
2	0.494	Background: 0.005	
3	0.494	Background: 0.006	
Fe(III)-Lactic acid-Activated Charcoal	0.834	0.1	16.6490 C
1	0.834	Background: 0.005	
2	0.833	Background: 0.005	
3	0.834	Background: 0.005	

Fe(III)-Activated Charcoal (Without Ligand)	0.796	0.1 15.8990 C
1	0.795	Background: 0.005
2	0.797	Background: 0.005
3	0.796	Background: 0.005

Test Results:

S.no	Test Parameters	Sample	Results
01.	Iron by AAS Analysis: (ppm)	Fe(III)-Lactic acid – Activated charcoal Sample	832.5ppm
02.	Iron by AAS Analysis: (ppm)	Fe(III)–Activated charcoal (control)	795.0 PPM

CONCLUSION:

From the current project it is clear that Lactic acid as a Chelating agent has played a vital role in adsorption of Fe (III) ions from aqueous solution by Activated charcoal and increases the adsorption up to **4.716%**. This aspect will be useful in designing the newer strategies of Heavy metal Remediation techniques using Natural Chelating Ligands as Facilitating agents in Metal Adsorption processes.

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Operator Name: Jyothi *Report Date:* 11-06-2022 17:40:20
Results File: E:\AAS System Data\2022\JUNE\11 06 2022\TARA 0064 2-6\Iron (Fe)1.SLR

General Parameters

Method : Iron (Fe) *Operator :* Jyothi *Instrument Mode:* Flame
Autosampler : None *Dilution:* None
Use SFI: No

Valid Method Signatures

11-06-2022 17:22:33 jyothi(M. Jyothi):DESKTOP-39TDEGC
Signed with Reason : Analysed by:
11-06-2022 17:23:24 parjanya(Parjanya):DESKTOP-39TDEGC
Signed with Reason : Approved by:

Method Audit Trail

11-06-2022 17:22:18 Jyothi(M. Jyothi):DESKTOP-39TDEGC
Record created
11-06-2022 17:22:33 jyothi(M. Jyothi):DESKTOP-39TDEGC
Signed with Reason : Analysed by:
11-06-2022 17:23:24 parjanya(Parjanya):DESKTOP-39TDEGC
Signed with Reason : Approved by:

Analysis Details

Analysis Name: Iron (Fe) 11-06-2022 *Spectrometer:* iCE 3000 AA01204906 v1.30
Operator Name: Jyothi

Lamp Information

Element(s)	**Serial Number**	**mA Hours**
Fe	n/a	n/a

Deuterium Lamp Hours: 68.34

Sequence Table

Shared Standards: Yes

Action	**Fe**
Calibration	✓
B.No.Group-II	✓
B.No.Group-III	✓
B.No.Group-IV	✓
B.No.Group-V	✓
B.No.Group-VI	✓

Sample Details

Nominal Mass: 1.0000

No.	**Sample Id**	**Sample Mass**	**Dilution Ratio**
1	B.No.Group-II	1.0000	1.0000
2	B.No.Group-III	1.0000	1.0000
3	B.No.Group-IV	1.0000	1.0000
4	B.No.Group-V	1.0000	1.0000
5	B.No.Group-VI	1.0000	1.0000

Valid Analysis Signatures

11-06-2022 17:38:41 jyothi(M. Jyothi):DESKTOP-39TDEGC
Signed with Reason : Analysed by:
11-06-2022 17:39:33 parjanya(Parjanya):DESKTOP-39TDEGC
Signed with Reason : Approved by:

Analysis Audit Trail

11-06-2022 17:30:16 Jyothi(M. Jyothi):DESKTOP-39TDEGC
Record created
11-06-2022 17:38:27 Jyothi(M. Jyothi):DESKTOP-39TDEGC
Error MD147 - Activity manually aborted by user.
11-06-2022 17:38:41 jyothi(M. Jyothi):DESKTOP-39TDEGC
Signed with Reason : Analysed by:
11-06-2022 17:39:33 parjanya(Parjanya):DESKTOP-39TDEGC
Signed with Reason : Approved by:

Spectrometer Parameters - Fe

Element: Fe
Measurement Mode: Absorbance
Wavelength: 248.3nm
Bandpass: 0.2nm
Lamp Current: 75%
Background Correction: D2
High Resolution: Off
Optimise Spectrometer Parameters: No
Signal Type: Continuous
Resamples: Fast
Number Of Resamples: 3
Measurement Time: 4.0secs
Flier Mode: No
Use RSD Test: No

Flame Parameters - Fe

Flame Type: Air-C2H2
Fuel Flow: 0.9L/min
Auxilary Oxidant: Off
Nebuliser Uptake: 4secs
Burner Stabilisation: 0mins
Optimise Fuel Flow: No
Burner Height: 7.0mm
Optimise Burner Height: No

Sampling Parameters - Fe

Sampling: None

Calibration Parameters - Fe

Calibration Mode: Normal
Line Fit: Linear
Use Stored Calibration: No
Concentration Units: mg/L
Scaled Units: mg/L
Scaling Factor: 1.0000
Acceptable Fit: 0.990
Rescale Limit: 10.0%
Failure Action: Flag and Continue

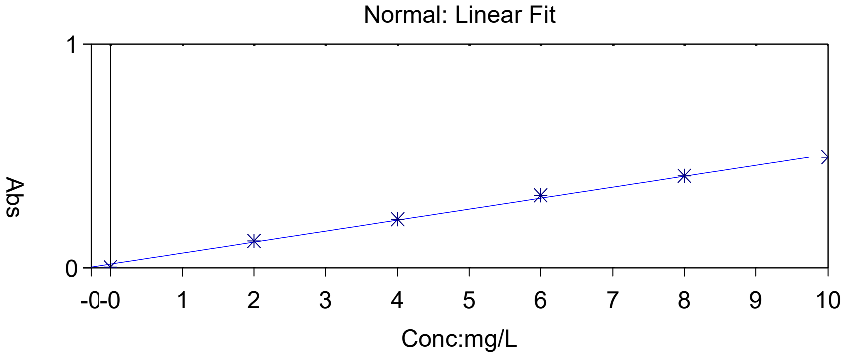
Standard 1	2.0000	Standard 4	8.0000
Standard 2	4.0000	Standard 5	10.0000
Standard 3	6.0000		

Element Audit Trail - Fe

No changes are recorded for this element

Solution Results - Fe

Y = 0.04924x + 0.0141
Fit: 0.9965
Characteristic Conc: 0.0894



Sample ID	Signal Abs	Rsd %	Conc mg/L	Corrected Conc mg/L
Fe Blank	**0.001**	**35.6**	**0.0000**	
1	0.001	Background: -0.003		11-06-2022 17:31:26
2	0.001	Background: -0.003		11-06-2022 17:31:30
3	0.000	Background: -0.003		11-06-2022 17:31:35
Fe Standard 1	**0.120**	**0.3**	**2.0000**	
1	0.120	Background: 0.003		11-06-2022 17:32:05
2	0.119	Background: 0.003		11-06-2022 17:32:09
3	0.120	Background: 0.003		11-06-2022 17:32:13
Fe Standard 2	**0.215**	**0.3**	**4.0000**	
1	0.215	Background: 0.004		11-06-2022 17:32:40
2	0.215	Background: 0.004		11-06-2022 17:32:45
3	0.216	Background: 0.004		11-06-2022 17:32:49
Fe Standard 3	**0.322**	**0.2**	**6.0000**	
1	0.323	Background: 0.005		11-06-2022 17:33:19
2	0.322	Background: 0.005		11-06-2022 17:33:23
3	0.322	Background: 0.005		11-06-2022 17:33:27
Fe Standard 4	**0.411**	**0.3**	**8.0000**	
1	0.410	Background: 0.005		11-06-2022 17:33:59
2	0.412	Background: 0.005		11-06-2022 17:34:03
3	0.412	Background: 0.005		11-06-2022 17:34:07

Operator Name: Jyothi *Report Date:* 11-06-2022 17:40:20

Results File: E:\AAS System Data\2022\JUNE\11 06 2022\TARA 0064 2-6\Iron (Fe)1.SLR

Solution Results - Fe

Sample ID	Signal	Rsd	Conc	Corrected Conc
	Abs	%	mg/L	mg/L
Fe Standard 5	**0.493**	**0.1**	**10.0000**	
1	0.494	Background: 0.005		11-06-2022 17:34:38
2	0.494	Background: 0.005		11-06-2022 17:34:42
3	0.493	Background: 0.006		11-06-2022 17:34:46
Fe B.No.Group-II	**0.834**	**0.1**	**16.6490 C**	**16.6490 C**
1	0.834	Background: 0.005		11-06-2022 17:35:14
2	0.833	Background: 0.005		11-06-2022 17:35:19
3	0.834	Background: 0.005		11-06-2022 17:35:23
Fe B.No.Group-III	**0.759**	**0.1**	**15.1286 C**	**15.1286 C**
1	0.758	Background: 0.003		11-06-2022 17:35:52
2	0.760	Background: 0.003		11-06-2022 17:35:56
3	0.758	Background: 0.004		11-06-2022 17:36:00
Fe B.No.Group-IV	**0.645**	**0.3**	**12.8189 C**	**12.8189 C**
1	0.647	Background: 0.003		11-06-2022 17:36:31
2	0.644	Background: 0.003		11-06-2022 17:36:35
3	0.645	Background: 0.003		11-06-2022 17:36:40
Fe B.No.Group-V	**0.809**	**0.2**	**16.1488 C**	**16.1488 C**
1	0.808	Background: 0.005		11-06-2022 17:37:11
2	0.809	Background: 0.005		11-06-2022 17:37:15
3	0.811	Background: 0.005		11-06-2022 17:37:20
Fe B.No.Group-VI	**0.711**	**0.3**	**14.1597 C**	**14.1597 C**
1	0.709	Background: 0.005		11-06-2022 17:37:55
2	0.713	Background: 0.005		11-06-2022 17:37:59
3	0.712	Background: 0.004		11-06-2022 17:38:03

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Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India,
Tel. : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com

DCA Approval No: 05/ML/TS/2020/G

CERTIFICATE OF ANALYSIS

MSL/QA/017-03/F07-00

Name & Address of the Customer:
Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-1
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-I
Mfg. date : NA
Exp. date : NA
Storage condition : To be stored at room Temperature
Batch size : NA
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	620ppm

Authorized Signatory

(Dr.R.Marayya)

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Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India,
Tel. : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com

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Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-2
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-II
Mfg. date : NA
Exp. date : NA
Batch size : NA
Storage condition : To be stored at room Temperature
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	832.5ppm

Authorized Signatory

(Dr.R.Marayya)

MART Specialities Lab. LLP
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Pragathi Nagar, Medhcal, Medchal-Malkajgiri - 500 055, Telangana, India,
Tel. : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com

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Name & Address of the Customer:
Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-3
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-III
Storage condition : To be stored at room Temperature
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Mfg. date : NA
Exp. date : NA
Batch size : NA

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	756.5ppm

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Name & Address of the Customer:
Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-4
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-IV
Mfg. date : NA
Exp. date : NA
Storage condition : To be stored at room Temperature
Batch size : NA
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	641ppm

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Tel. : 85007 98350, 81423 98350 & 98481 98350 **Mail**:martspecialities@gmail.com

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MSL/QA/017-03/F07-00

Name & Address of the Customer:
Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-5
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-V
Storage condition : To be stored at room Temperature
Mfg. date : NA
Exp. date : NA
Batch size : NA
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	807.5ppm

Authorized Signatory

(Dr.R.Marayya)

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Tara Government College
Prashanth Nagar Colony, Balajinagar
Sangareddy Telangana. 502000
Contact Person: Dr. Abhijeet
Contact Number :9502344392

Reference / Report No. : MSL/2022/JUNE/TARA/0064-6
Sample Received Date : 11/06/2022
Report Date : 13/06/2022

DETAILS OF THE SAMPLE

Sample Name : NA
Name of the Manufacturer : NA
Batch no : Group-V1
Mfg. date : NA
Exp. date : NA
Batch size : NA
Storage condition : To be stored at room Temperature
Room Temperature : 25°C±3°C
Quantity Received : 4gm
Tests Required : Iron by AAS Analysis.
Method : NA
Analysis Starting Date : 11/06/2022
Analysis Completion Date : 11/06/2022
Mfg. License No. : Not provided
A.R.NO : NA
Remark : Sample analyzed as received

Test Results

S.No.	Test Parameter	Result
01.	**Iron by AAS Analysis: (ppm)**	708ppm

Authorized Signatory

(Dr.R.Marayya)