A STUDY ON NATURAL ADSORBENTS FOR THE REMOVAL OF CHLORIDE ION IN WATER

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Abstract

Next to air, the most important requirement for human to exist is water, which is the gift of nature. With growing population, the demand for drinking water is increasing day by day and hence preventive measures are to be taken to prevent the pollution and of water. Chlorides contamination are generally present in water in the form of NaCl. Chloride in concentration above 600mg/l tends to give water a salty taste. WHO specifies Highest desirable concentration of chloride in portable water must be 200ppm. Chloride attack is one of the most important aspects for consideration when we deal with the durability of concrete. Chloride attack primarily induces corrosion, which is responsible for 40% of the failure of structure .As per IS46:2000 the water used for mixing and curing concrete should comprise a chloride content with a permissible limit of 2000mg/l for plain concrete and 500mg/l for reinforced concrete work. In industries there are various methods adopted to produce potable water, of which, the adsorption process is a widely used phenomenon. Here for the removal of chlorine we are using E.crassipes (A natural adsorbent) and Amberlite (a synthetic adsorbent). The objective of our project is to find the adsorption capacity of E.crassipes and Amberlite for the removal of chloride ion in water. The adsorption capacity is found out by Batch studies which include Effect of dosage, Effect of pH, Effect of initial concentration and Effect of contact time. And adsorption capacity is found out theoretically by Langmuir adsorption isotherm and freundlich adsorption isotherm.

Key Words: EICHHORNIA CRASSIPES, Chloride ions, ion exchange, natural adsorbent

1. Introduction

1.1 General

There are various purification methods to make water potable, as required by the public water supply scheme. The water required for domestic consumption should possess a high degree of purity and should be free from suspended impurities, bacteria, etc. Thus, the drinking water must meet the highest standard of purity, which is possible by maintaining the various constituent concentrations in water within the permissible limits. In industries there are various methods adopted to produce potable water, of which, the adsorption process is a widely used phenomenon.

1.2ADSORPTION

1.2.1 ADSORPTION PROCESS

Adsorption is a mass transfer process that occurs when a gas or a liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. It is a surface phenomenon. Adsorption is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. In the case of adsorption, the adsorbate is attached to the surface of the adsorbent.

1.2.2 ADSORPTION TYPES

Adsorption material is generally classified as exhibiting physisorption or chemisorption.

i) **Physisorption** or physical adsorption is a type of adsorption in which the adsorbate adheres to the surface only through Vander Waals (weak intermolecular) interactions, which are also responsible for the non-ideal behavior of real gases. Physical adsorption is a reversible phenomenon and it is possible to remove the contaminants from adsorbent surface.

ii) **Chemisorption** is a type of adsorption, whereby a molecule adheres to a surface through the formation of a chemical bond, as opposed to the Vander Waals forces, which cause physisorption. It occurs as a result of chemical reaction between solute molecules and adsorbent surface. This type occurs on specific sites like the corners of the micro crystallites of an adsorbent. Chemisorption is usually irreversible and separation of the adsorbent from solute is difficult.

1.2.3 EFFECTIVE USE OF ADSORPTION

Adsorption process is utilized to remove soluble organic compounds from waste water. Through this process, contaminant from waste water is transferred to the surface of adsorbent material. Adsorption occurs primarily due to physisorption. It is used in treating waste water containing coloured chemicals, herbicides, pesticides, certain petrochemicals and heavy metals.

Adsorption is operative in most natural, physical, biological and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins for water purification. The most common industrial adsorbents are,

- a. Activated carbon.
- b. Silica gel.
- c. Alumina.

1.3 ADSORBENT

1.3.1 OVERVIEW

Adsorbents are highly porous materials and adsorption occurs primarily on the walls of pores or at specific sites inside the particles. The pores are generally small and internal surface area ranges from 500-1000m²/gm.Adsorption continues until all available adsorption sites are covered. Commercially used adsorbents are, activated carbon, diatomaceous earth and certain type of clay.

1.3.2 ACTIVATED CARBON

Activated carbon is a magnificent adsorbent compared to silica gel and alumina and is used widely. It is also called activated charcoal or activated coal. It is a material with exceptionally high surface area (1gm of carbon has a surface area of $500m^2$).

Activated carbon is produced by roasting organic material to decompose it to granules of carbon. Common sources are coconut shell, ground nut shell, coffee husk, bone charcoal, saw dust, seed of plants etc. Silica gel is a matrix of hydrated silicon dioxide. Alumina is mined or precipitated aluminum oxide and hydroxide.

1.4 CHLORIDE

1.4.1 FORMS OF CHLORIDE

Chlorides are generally present in water in the form of NaCl (common salt) and may be due to leaching of marine sedimentary deposits, pollution from sea water, brine or industrial and domestic wastes, etc.

1.4.2 EFFECTS OF CHLORIDE

Chloride in concentration above 600mg/l tends to give water a salty taste. The concentration of chloride content above 200mg/l is considered objectionable. Presence of high quantity of chloride content in water resources indicates pollution due to human and industrial wastes and also from the earthen rocks in the subsurface.

1.4.3ENVIRONMENTAL SIGNIFICANCE OF CHLORIDE

There is no known evidence that chlorides constitute any human hazard. The concentration of chloride content above 250mg/l is undesirable. For this reason, chlorides are generally limited to 250mg/l in supplies intended for public use. Presence of chloride increases the hardness of water, thereby produces a salty taste of water, which is not fit for drinking.

2. EXPERIMENTS AND TESTING

2.1 GENERAL

Chloride concentration was determined by argentometric method. The experiment is done with two different adsorbents

- 1) Water Hyacinth (Eichhornia crassipes)
- 2) Anion exchange resin(amberlite)

The preparation of the adsorbent from water hyacinth includes various procedures like acid activation and thermal activation. Amberlite is used without any activation.

2.2ACTIVATED CARBON - WATER HYACINTH AS AN ADSORBENT

Water Hyacinth (Eichhornia crassipes), an aggressive exotic aquatic plant, is native to Brazil but now occurs globally. E. crassipes is a very hardy and persistent species that is established in a wide range of aquatic habitats. It is a highly competitive plant that is capable of rapid growth and spread. It is a fast growing free-floating noxious weed that has attracted worldwide attention due to its fast spread and congested growth, which lead to serious problems in navigation, irrigation, and power generation. On the other hand, when looked from a resource angle, it appears to be a valuable resource with several unique properties. This plant has the ability to remove heavy metal contaminants from the surrounding water through biosorption.As a result, research activity concerning control (especially biological control) and utilization (especially wastewater treatment or phytoremediation) of water hyacinth has boomed up in the last few decades. Investigations on biogas/compost production from water hyacinth have also come up very well, mainly from few research groups in India. Since this plant is easily available and has the ability to adsorb the contaminants from water, it is chosen for our study. We have studied in detail about the

usage of Water hyacinth as an adsorbent for the removal of chloride ion.



Figure2.1:EICHHORNIA CRASSIPES(WATER HYACINTH PLANT) HYACINTH

2.2.1 IMPACTS AND THREATS POSED BY WATER HYACINTH

E. crassipes is a highly competitive plant that is capable of rapid growth and spread.E.crassipes can displace native species, reduce biodiversity, limit recreation, diminish aesthetic value, and decrease water quality and flow.

- Dense floating rafts of Water Hyacinth can form on the water's surface, restricting light to the complete exclusion of other native plants, and decreasing the air exchange between the water's surface and the atmosphere.
- Thick floating mats can prevent fishing, boating, swimming and other activities and the loss of recreational and aesthetic value can cause a decline in surrounding lake property value.
- Algae, a major component of the base of the food chain, can be shaded out by dense mats of Water Hyacinth. The resulting decline in algae can disrupt the entire food web in a water body.
- E. crassipes may form dense single species stands that often do not provide ideal habitat or food for native wildlife and may limit access to the

water for some species. These native wildlife populations may be forced to relocate or perish, ultimately resulting in a loss of biodiversity and a disruption in the balance of the ecosystem.

- Decomposition of E. crassipes can create anoxic (low oxygen) conditions in the water which may result in fish kills.
- Dense stands trap sediments, slow water flow in irrigation channels and waterways and may provide a breeding ground for mosquitoes.
- Sediment levels increase with increasing E. crassipes abundance.

For these threats in the environment, the water hyacinth can be used as an adsorbent in the removal of ions.

2.2.2 PREPARATION OF ADSORBENT:

The adsorbent, for this experiment, is prepared in the form of activated carbon. As the raw material for the preparation of activated carbon is an aquatic weed, the production of this carbon is economically feasible.

Step 1: The water hyacinth plants are collected from a nearby pond. It is then sundried for about one week.

Step 2: The plants are then washed several times with clean water to remove the dirt, mud, mosses, etc.

Step 3: Subsequent washing with distilled and double distilled water are also done to remove the tedious material. It is then dried.

Step 4: <u>Acid Activation</u>: About 25 grams of this material is treated with 20ml of concentrated sulphuric acid and the charred material was kept overnight. The charred material is heated in an oven at $100\Box C$ for about 4-6 hours. This is cooled and washed with distilled water to remove any trace of acid, so that the pH ranges between 6 and 8, which is suitable for the determination of chloride by argentometric method. The main

purpose of acid activation is to ensure a highly protonated surface on the adsorbent material. Higher adsorption rate of chloride in the acidic range can be explained by the surface charge of the adsorbent. By acid treatment the surface of the adsorbent is highly protonated and hence more chloride can be attracted to the surface of the adsorbent. High chloride sorption rate in the acidic medium is contributed due to strong columbic forces between positively charged surface and negatively charged chloride ions. Hence acid activation ensures higher efficiency in the removal of chloride ions.

Step 5: <u>Thermal Activation:</u> Thermal activation is done in order to prepare activated carbon with a definite pore structure possessing efficient adsorption capacity. The activation provides a material of high surface area and strong sorption capacity towards various adsorbates. A large surface area increases the adsorption rate, which is ensured only due to thermal activation.

2.3 AMBERLITE

The **AMBERLITE** products have unique capacities for the biopharmaceutical industry in the purification, isolation, and decolorization of organic or aqueous solutions derived from natural product extraction, synthesis or from fermentation. Amberlite is a synthetic resin



FIGURE 2.2: ION-EXCHANGE RESIN BEADS

Synthetic resin is insoluble substances containing loosely held ions which are able to be exchanged with other ions in solutions which come in contact with them. These exchanges take place without any physical alteration to the resin.

Synthetic ion exchange materials based on coal and phenolic resins were first introduced for industrial use during the 1930.s. A few years' later resins consisting of polystyrene with sulphonate groups to form cation exchangers or amine groups to form anion exchangers were developed. These two kinds of resin are still the most commonly used resins today.

2.3.1 PROPERTIES OF AMBERLITE

Physical fo	rm	: Pale yellow
translucent	spherical be	ads

Matrix benzene co polymer	: Styrene divinyl
Functional group ammonium	: Trimethyl

Moisture holding capacity: 49%-60%

Particle size

Uniform coefficient:< 1.6

Harmonic mean size: 0.6mm to 0.75 mm

Operating conditions

Maximum operating temperature: 60° C

Minimum bed depth	: 700mm
Regeneration	: Treating
with NaOH	

2.3.2 HOW RESINS WORK

The resins are prepared as spherical beads 0.5 to 1.0 mm in diameter,

usually white, yellowish or reddish, fabricated from an organic polymer substrate. These appear solid even under the microscope, but on a molecular scale the structure is quite open. This means that a solution passed down a resin bed can flow through the cross linked polymer, bringing it into intimate contact with the exchange sites.

The material has highly developed structure of pores on the surface of which is sites with easily trapped and released ions. The trapping of ions takes place only with simultaneous releasing of other ions, hence the name ion-exchange.

2.3.3 USES OF RESIN

Resins are widely used in different separation, purification and decontamination processes. A bed of resin can be used either to remove unwanted ions from a solution passed through it or to accumulate a valuable mineral from the water which can later be recovered from the resin. Examples of the removal of unwanted ions are the removal of heavy metals from metal trade wastes and the removal of salts from fruit juices.

Strong cation resins in the hydrogen form are used for the hydrolysis of starch and sucrose. Resins also find many uses in the laboratory where the chemist's ingenuity is less constrained by economic considerations. They can be used to remove interfering ions during analysis or to accumulate trace quantities of ions from dilute solutions after which they can be concentrated into a small volume by elution.

A cation resin in the hydrogen form can be used to determine the total concentration of ions in a mixture of salts. The sample passing through a column is converted to the equivalent quantity of acid and the amount readily found by titration. One of the earliest applications of ion exchange was the separation of rare earth elements during the 1940.s. These metals occur naturally as mixtures and have almost identical chemical properties. The equilibrium quotients for cationic resin were found to vary sufficiently for separation to be achieved chromatographically by adding a solution of the mixture to a resin column and eluting the metals with an acid wash. Besides being made as bead-shaped materials, ion-exchange resins are produced as membranes that allow passage of ions, in electro dialysis.

2.3.4 ENVIRONMENTAL IMPLICATIONS

The resin in industrial use is generally regenerated every 12-48 hours. The exchange is made more efficient by introducing the regenerant at the bottom of the resin column and passing it upwards through the bed. The waste water for disposal after regeneration contains all the minerals removed from the water plus salt from the spent regenerants. These are concentrated into a volume equivalent to 1-5% of the treated water throughout. Disposal is not usually a problem as the load on the waste treatment systems is low as compared with that, from many other industrial processes.

2.4DETERMINATION OF CHLORIDE ION:

Method used: Argentometric method Principle:

Chloride ion is determined by titration with standard silver nitrate solution in which silver chloride is precipitated first, due to the reaction of silver nitrate with chloride ions. The end of titration is indicated by formation of red silver chromate from excess AgNO₃ and potassium chromate used as an indicator in neutral to slightly alkaline solution.

 $AgNO_3 + Cl^- \rightarrow AgCl + NO_3^-$

 $2AgNO_{3+}K_2CrO_4 \rightarrow Ag_2CrO_4+2KNO_3$

Interference:

The substances found in potable water do not interfere. Bromide, iodide and will titrate chloride. cyanide as Orthophosohate in excess 25 mg/lof interfere.Sulphide, sulphite, thiosulphate and thiocyanate ions also interfere. However, these interferences can be eliminated the addition of hydrogen peroxide.

Apparatus required:

- Burette
- Pipettes
- Conical Flask
- Reagents used:
- Chloride free distilled water.
- Standard Silver nitrate solution (0.0141N): Take 2.395g of silver nitrate and dilute to 1000ml with distilled water.
- Potassium Chromate indicator.
- Sodium chloride solution. (sample solution):Take suitable gram of NaCl salt and dilute to 1000 ml using distilled water to achieve required normality.

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Procedure:

1. Take 50ml of the sample in a conical flask

2. Adjust its pH to be between 7 and 8 either with sulphuric acid or sodium hydroxide solution. Otherwise, AgOH is formed at high pH levels or CrO_4^{-2} is converted to $\text{Cr}_2\text{O}_7^{-2}$ at low pH levels.

3. Add 1ml of potassium chromate indicator to get a light yellow colour.4. Titrate with standard silver nitrate solution till colour change from yellow to brick red.

5. Note the volume of silver nitrate added. (A)

6. If more quantity of potassium chromate is added, Ag_2CrO_4 may form too soon.

7. For better accuracy, titrate distilled water in the same manner.8. Note the volume of silver nitrate added for distilled water (B).

Calculations:

Chloride content ($\underline{mg/l}$) =(A-B) x normality of AgNO₃ x 35.45 x 1000

Volume of

sample taken

, oranic or

Where: A = ml titration for sample, B = ml titration for blank.

2.5 BATCH STUDIES

Batch adsorption experiments were carried out by subjecting various doses of the adsorbent to different concentrations of chloride samples. The adsorption efficiency, under various experimental conditions, is arrived at by titration procedure.

The batch study is conducted according to the following steps:

- A known amount of the adsorbent is taken in a conical flask.
- The sample solution of known concentration is mixed thoroughly with the adsorbent.
- The conical flask containing the mixture is shaken vigorously in a rotary shaker at 160 rpm for 4-6 hours.
- The mixture is then filtered using whatman filter paper (No.42).
- The filtrate containing the residual concentration of chlorine is determined by argentometric method.
- Hence the efficiency in the removal of chloride is found out from the initial and final concentration of chlorine in the sample.
- The following steps are carried out under different experimental conditions and the graphs are plotted.

2.5.1 EFFECT OF DOSAGE:Various dosage of adsorbent was shaken with 50ml of known concentration. The samples were

collected after a particular time. The treated chloride solution was filtered with a whatman filter paper no: 42 and the filtrate were titrated against silver nitrate to determine the amount of chloride adsorbed.

2.5.2 EFFECT OF INITIAL CONCENTRATI:The initial concentration of chloride was varied from 10ppm to 300ppm .The adsorbent were added and the effective removal of chloride was determined.

2.5.3 EFFECT OF pH:The pH of the chloride solution was varied from 2 to 14. The adsorbent were added and the effective removal of chloride was determined.

2.5.4 EFFECT OF CONTACT TIME:The adsorbent and the chloride solution are shaken in the wrist action shaker for various contact time from 1hr to 6hours and the effective removal of chloride was determined.

2.6

ADSORPTION

ISOTHERM:Adsorption is usually described through isotherms, that is, the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. The Langmuir adsorption model is the most common model used to quantify the amount of adsorbate adsorbed on an adsorbent as a function of partial pressure or concentration at a given temperature. The first mathematical fit to an isotherm was published by Freundlich and Küster (1894) and is a empirical formula for purely gaseous adsorbate.

$$\frac{x}{m} = kP^{\frac{1}{n}}$$

Where, x is the quantity adsorbed, m is the mass of the adsorbent, P is the pressure of adsorbate and k and n are empirical constants for each adsorbent-adsorbate pair at a given temperature. The function has an asymptotic maximum as pressure increases without bound. As the temperature increases, the constants k and n change to reflect the empirical observation that the quantity adsorbed rises more slowly and higher pressures are required to saturate the surface.

2.6.1 COMPUTATION OF THE ISOTHERM:Experimental evaluation of isotherm constants is a standard practice for judging the feasibility of an adsorbing system. The equilibrium adsorption data were fitted to both Langmuir isotherm as well as freundlich adsorption model

$$X/M = [ab Ce]/1 + aCe$$

The **Langmuir** isotherm can be linearised to the following equation.

Ce/X/M = [1/ab] + [Ce/b]

Where Ce (mg/l)

And X/M (mg/g) is concentration of chloride and amount of chloride adsorbed at equilibrium. From the linearised form of the Langmuir isotherm, the isotherm constants 'a'- Constants which increase with increasing molecular size R_L – Amount adsorbed to form a complete monolayer on the surface The essential features of the Langmuir isotherm can be expressed in terms of dimensionless separation factor R_L

 $R_{L} = 1/(1+bXC_{i})$

Where, C_i- Initial Chloride Concentration

Separation factor R_L indicates the isotherm shape accordingly.

R_L>1, Unfavorable

 $R_L = 1$, Linear

R_L lies between 0 and 1, Favorable

R_L=0, Irreversible.

The **Freundlich** isotherm can be linearised to the following equation

$$\log (X/M) = \log K_f + (1/n) \log Ce$$

 $K_{\rm f}$ – constant which is a measure of adsorption capacity and 'n' is a measure of adsorption intensity. From the linearised form of freundlich isotherm the isotherm constants $K_{\rm f}$ and n can be determined using linear regression.

3. RESULTS AND DISCUSSIONS

3.1 GENERAL: Both adsorbents work were studied separately and the results of both were compared as discussions.

3.2 EICHHORNIA CRASSIPES

3.2.1 BATCH STUDIES

The adsorbent in the form of activated carbon was subjected to different environmental conditions like varying the adsorbent dose, contact time, chloride content, pH, temperature, etc.

3.2.1.1 EFFECT OF DOSAGE

Various dosage of adsorbent was varied from 1g to 6g and shaken with 50ml of constant concentration of 100mg/l.The samples were collected after a particular time. The treated chloride solution was filtered with a whatman filter paper no: 42 and the filtrate were titrated against silver nitrate to determine the amount of chloride adsorbed.

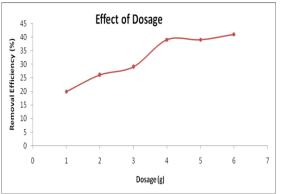
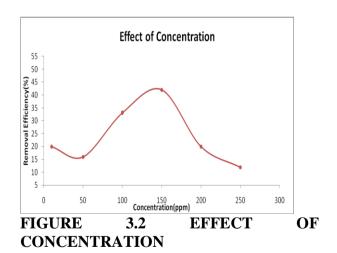


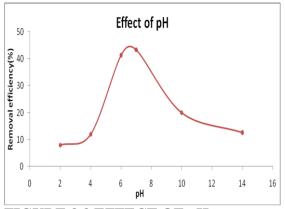
FIGURE 3.1 EFFECT OF DOSAGE

3.2.1.2EFFECT OF INITIAL CONCENTRATION:The initial concentration of chloride was varied from 10ppm to 250ppm with a constant dosage of 4g of the adsorbent and the effective removal of chloride was determined.



3.2.1.3 EFFECT OF PH

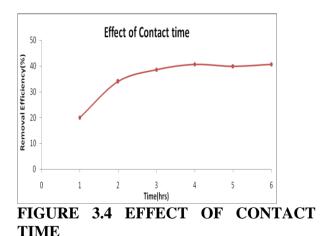
The pH of the chloride solution was varied from 2 to 14 with a constant concentration of chloride of 150ppm and a constant dosage of 4g.





3.2.1.4 EFFECT OF CONTACT TIME

The adsorbent and the chloride solution are shaken in the wrist action shaker for various contact time from 1hr to 6hours. A constant concentration of chloride of 150ppm and a constant dosage of 4g were used.



3.3 AMBERLITE

3.3.1 BATCH STUDIES

Similar to the batch studies of water hyacinth batch adsorption experiments for Amberlite(a synthetic resin) were carried out by subjecting various doses of the adsorbent to different concentrations of chloride samples, temperature, contact time, etc.

3.3.1.1 EFFECT OF DOSAGE

Various dosage of adsorbent was varied from 0.5g to 3.5g shaken with 50ml of constant concentration of 100mg/l. The samples were collected after a particular time. The treated chloride solution was filtered with a watt man filter paper no: 42 and the filtrate were titrated against silver nitrate to determine the amount of chloride adsorbed. Based on the increase in dosage, the efficiency in the removal chloride is hence determined.

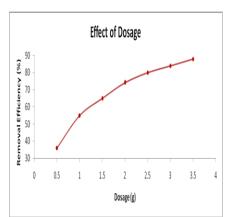


FIGURE 3.5 EFFECT OF DOSAGE

3.3.1.2 EFFECT OF INITIAL CONCENTRATION

The initial concentration of chloride was varied from 30ppm to 300ppm with a constant dosage of 3g of the adsorbent and the effective removal of chloride was determined.

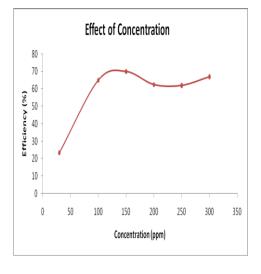


FIGURE 3.6 EFFECT CONCENTRATION

OF

3.3.1.3 EFFECT OF pH

The pH of the chloride solution was varied from 2 to 14 with a constant concentration of chloride of 150ppm and a constant dosage of 3g.

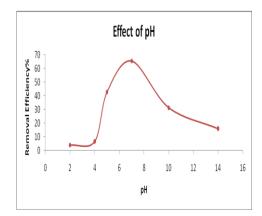


FIGURE 3.7 EFFECT OF pH

3.3.1.4 EFFECT OF CONTACT TIME

The adsorbent and the chloride solution are shaken in the wrist action shaker for various contact time from 1hr to 6hours. A constant concentration of chloride of 150ppm and a constant dosage of 3g were used.

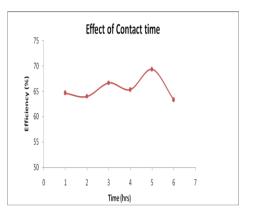


FIGURE 3.8 EFFECT OF CONTACT TIME

4. CONCLUSION

The Adsorption capacity of Water hyacinth and Amberlite were studied using chloride solution. Amberlite is better adsorbent compared to water hyacinth as the former shows best fit for both Langmuir and Freundlich isotherms.Water hyacinth showed unfavorable adsorption with R_L =2.91 which is greater than 1 and with Amberlite it shows R_L =0.07 which is less than 1

Future Scope:

Batch studies can be done by increasing the adsorbent dosage and the concentration.

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