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Synthesis and characterization of chitosan – Iron oxide nano particle for Batch Adsorption of lead (II) ions from aqueous solutions.

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ABSTRACT

A novel chitosan was designed to prepare deacetylation and deproteination method.Chitosan was mixed with Fe_3O_4 using formaldehyde to form chitosan –Fe_3O_4 nanoparticles.The prepared products were charecterised by using SEM,EDAX,TGA. SEM shows wide range of porosity that could achieve high lead (ll) adsorption .EDAX is used to confirm lead present after adsorption.TGA is used to determine total weight loss 17%.The influence of P^H adsorbent dosage and initial metal ion concentration on removal of lead (II) was investigated. The adsorption efficiency was found to be P^H dependent and 93% lead (II) removal was observed at optimum P^H6. Results showed that the maximum adsorbent capacity was at dosage of 3.5g and equilibrium time achieved at 360 minutes.

Keywords: Chitosan, Lead (II) Nitrate, SEM, EDAX, etc

Introduction

The presence and high concentration of heavy metal ions in various water resources can be adverse to the environment and public health. Among all the heavy metal ions, special attention has been given to Pb (II) ions contamination in water. Lead metal is listed second on the ATSDR (Agency of toxic substances and Disease Registry) list of top 20 hazardous substances. Pb (II) ions can be found in effluents from battery recycling plants, lead mining and electronic assembly plants. Lead elucidates destructive effects on human nervous system, blood circulation system, kidneys, reproductive system (1) and is highly toxic and carcinogenic even at low concentration (2)

According to WHO, the maximum permissible limit (MPL) of lead in drinking water is 50 Pb in 1995, which is decreased to 10 Pb in 2010. The wide usage of Pb (II) in various industries has triggered the necessities of developing an efficient method to remove this heavy metal ion from waste water. Many conventional methods are known for lead removal from water namely chemical precipitation (3) membrance separation, ion exchange (4,5) coagulation, reverse osmosis, evaporation (6) and adsorption.

Adsorption, an effective separation process for a wide variety of applications, is now reorganized as an effective and economical method for the removal of pollutants from waste water. The most widely used adsorbent is activated carbon but it is quite expensive and possibly, no cost effective in the treatment of large waste water volumes. That is why in the past few years, considerable attention has paid to low-cost biosorbents, as an alternative to reduce the cost of adsorption systems. (7,8)

Many naturally occurring materials have been investigated for assessing their suitability in controlling water pollution peat (9-10), lignin, gungi, (12-13) are well known as low-cost biosorbent for removing lead (II) from aqueous solutions. Another interesting example of biosorbent is chitosan .Several studies have shown that chitosan is very efficient in removing various toxic and strategic metals, such as cadmium, mercury, molybdenum Uranium, Vanadium, platinum and palladium (14,15)

Chitosan a hydrophilic natural polymer produced by alkaline deacetylation of chitin, obtained mainly by extraction from shrimp and crab shells is the most abundant biopolymer occuring in nature, after cellulose (16). It is characterized by a high content of nitrogen present as amino groups and is capable of absorbing the metal ions through several mechanisms – including chemical interactions such as chelations, electrostatic interactions or ion exchange. The interaction type depends (17, 18) on the metal ion, its chemistry and initial P^H of the solution

The objective of the present investigation is to find out the optimum Conditions for removing the lead (II) ions from aqueous solutions through adsorption using Iron doped chitosan powder.

MATERIALS AND METHODS

All chemicals used are of analytical reagent grade . A stock solution of Pb (II) (1000ppm) was prepared by dissolving the calculated quantity of $Pb(NO_3)_2$ in deionized water.

Adsorbent

Synthesised Iron oxide nano particles were dried and powdered. About 200 mg of 85% deacetylated chitosan powder and iron oxide were made in to a gel using 2-5 ml of formaldehyale. The mixture was stirred in a magnetic stirrer for 1 hour. Finally chitosan coated Iron oxide nanoparticle was obtained.(19)

Batch experiments

Batch adsorption experiments of the lead ion adsorption by chitosan encapsulated Iron oxide adsorbents were carried out at room temperature by shaking a series of bottles each containing the desired quantity of the adsorbent in a predetermined concentration of heavy metal solution samples were withdrawn at different time intervals. The supernant solution was separated by filtration and analyzed for remaining heavy metal content. The percentage removal of heavy metal from solution was calculated by the following equation.

% Adsorption=C_0-C_e/Cox100---(1)

Co - initial concentration of heavy metal ion in the solution (mg/l)

Ce - final concentration of heavy metal ion in the solution (mg/l)

$q_e = C_0 - C_e * V/m$ -----------------(2)

m - mass of the adsorbent

The Freundlich isotherm model as in Freundlich(1926) is defined as

$q_e = K_f C_e^{1/n}$ -----------------------(3)

SEM

A scanning electron Microscope (SEM) scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and Composition. Chitosan coated Iron oxide prepared from shrimp shell waste was examined by SEM having a magnification range 1um and accelarating voltage 20kv.

EDAX

Energy dispersive X-ray spectroscopy is used to detect elemental analysis (or) chemical characterization of a sample using oxford instrument.

TGA

TGA is a technique that measures mass change in a sample and it is used to detect evaporation ,decomposition ,oxidation and other effects of temperature change that cause mass change. A perkin –Elmer model TGA- thermo gravimetric system with a micro processor driven temperature control unit and a TGA data station was used.

RESULTS AND DISCUSSION

SEM

Scanning Electron Microscope (SEM) images shown in fig.1a+1b) revealed the nature of the morphology of the modified adsorbent before and after adsorption. Fig 1a) shows unoccupied pores on the before adsorption .Fig 1b) shows the morphology of the adsorbent after the flaky like structure of pb(II)ions. As seen in Fig 1b the outer pores are covered by Pb(II)ions and the pores are no longer visible. This is an evidence for the adsorption of Pb(II) metal ions on the surface of the adsorbent material.(20)

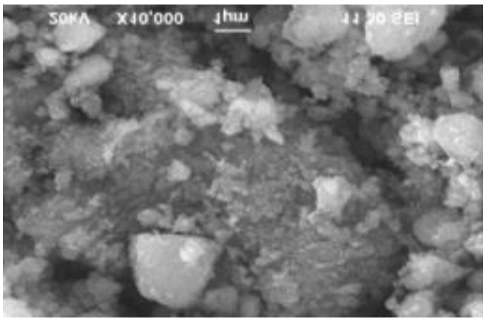


Fig1a)SEM image of the modified adsorbent before adsorption

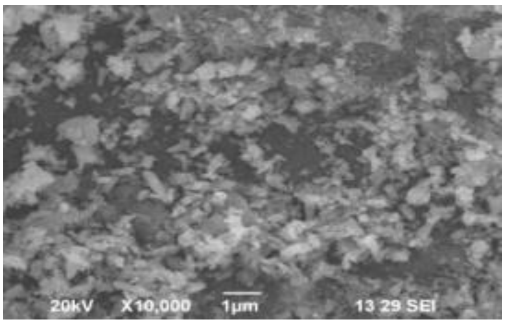
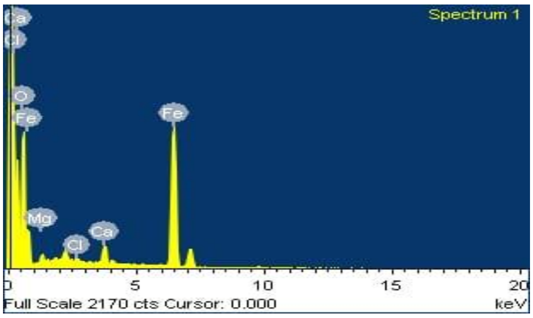


Fig 1b) SEM of the modified adsorbent after adsorption

EDAX

EDAX image shows that the synthesized nanoparticles indicate the presence of Pb in the deposited films



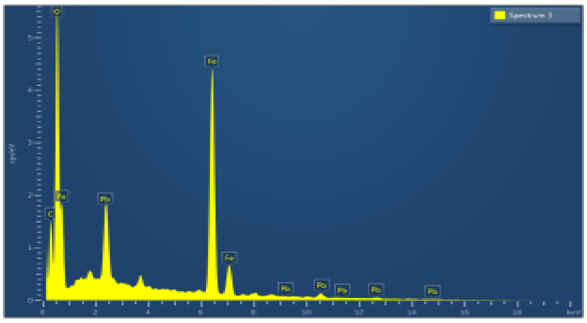


Fig2a)Energy dispersive x-ray spectroscopy before adsorption	Fig2b)Energy dispersive x-ray spectroscopy after adsorption

THERMO GRAVIMETRIC ANALYSIS

Thermo gravimetric Analysis is used to find the thermal stability and to determine the decomposition temperature of particles. The mass of the samples was generally 30 mg. The temperature range studied was 100ºC at the heating rate of 10ºC/min under nitrogen temperature. The mass of the sample pan was contionously recorded as a function of temperature Fig (3)show the results of thermogravimmetric analysis of chitosan-iron oxide nanoparticles. Two weight loses were observed in the TGA curve. The first stage ranges between 100-340ºC. This is due residue to moisture vapourization. The second stage is due to the decomposition of iron oxide chitosan and liberation of amine group at 400-800ºC . The total weight loss is 17%.

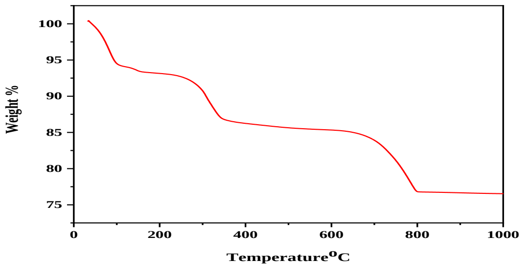


Fig : 3 Thermo gravimetric Analysis Effect of adsorbent dosage on lead removal

Adsorbent dosage effect on lead (ll)sorption was shown in(Fig -4---).Experimental studies were carried out by varying the adsorbent dose from 0.5 to 3.5g using of 20 mg/l of lead(ll)ion concentration at optimum pH 6 using contact time360 minutes.Results shown that the adsorption of lead (ll) ions increased with increased of adsorbent dose and attained the maximum at dosage of 2.5 g adsorbent.After this adsorbent dosethere no more adsorption efficiency.(21) It can be concluded that increasing the adsorbent dosage increases the available binding sites.Thus more surface area is available for adsorption ,there by increasing the lead percentage removal from the solution.(22).Maximum percentage removal of lead (ll)was found to be 90.89% at 3.5g.

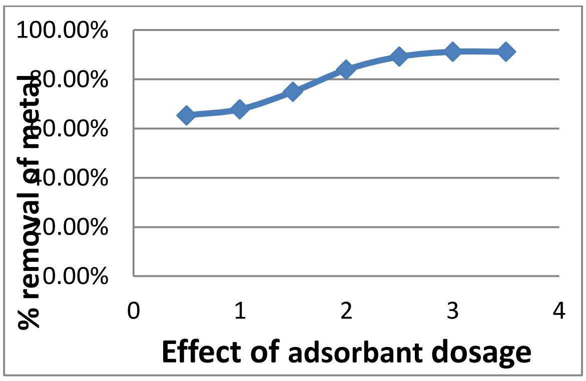


Fig : 4 Removal of metal against adsorbent dosage Effect of initial metal concentration

The effect of initial concentration in the range 10mg/l to 70 mg/l on adsorption of lead (ll)was found by keeping the remaining parameters like initial metal concentration,PH and adsorbent dosage are shown in fig 5.From the results it can be concluded that increase initial metal concentration, the percentage removal of the metal ion from solution because at higher concentration metal ions diffuse to the adsorbent surface and the hydrolysed ions diffuse at a slower rate, thus decreasing the percentage removal. (23) Also at higher metal ion concentration to adsorbent ratio energy sites get saturated resulting in lower percentage removal of metal ions. Maximum percentage removal decreased from 10 mg/l to 60 mg/l (24) Maximum percentage removal of lead (ll)was found to be 90.89% at 3.5g

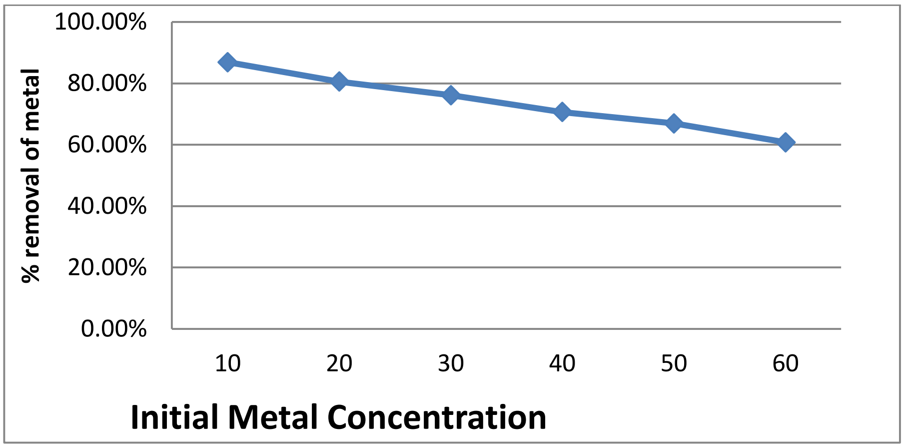


Fig : 5 Removal of metal against concentration Effect of P^H

The P^H of the aqueous phase has a considerable impact on the adsorption capacity.Depending on the P^H,heavy metal generate different ionic species .The majority of heavy metals tend to precipitate at higher P^H levels.Below P^H7 Pb^{2+} is known to be the predominant species and above P^H 8 Pb $(OH)_2$ becomes the dominant species there by leading to precipitation.(25)

The effect of P^H on percentage removal of Pb (II) by chitosan Iron oxide adsorbent is shown in Fig 6. It is clear from figure that the percentage removal of pb(II) increases slowly with increasing P^H from 2 to 8, and after that P^H drops slowly. The maximum percentage removal of pb (II) by chitosan- Iron oxide is 93.15% at P^H 6.

The optimum P^H value for adsorption of pb (II) by chitosan – Iron oxide is found to be 6.

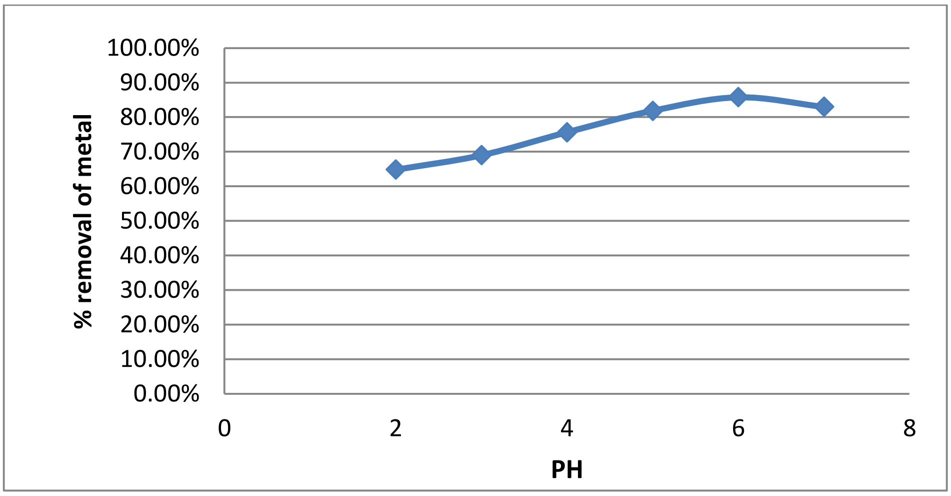


Fig : 6 Effect of P^H on adsorpition of lead

Adsorption Isotherm

q_e is calculated from eqn(2) and by using the data in batch study. To obtain a linear relationship a plot of values for $1/q_e$ (y axis) vs values of $1/c_e$ (x axis) is plotted.

Figure-(8,10,12) shows the relationship between $1/q_e$ and $1/c_e$ for chitosan-Fe_3O_4 nano particles from this one can determine the value of K from the slope of the best fit line and the value from the intercept

The Freundlich isotherm can be transformed to a linear equation by taking the (Ln) of both sides of equation_(26)

When Ln (q_e) is plotted on the Y axis and Ln (c_e) on the x axis the adsorption data obeyed Freundlich adsorption isotherm , The plot of Ln(q_e) Vs Ln(c_e) from Fig-7,9,11, is Linear

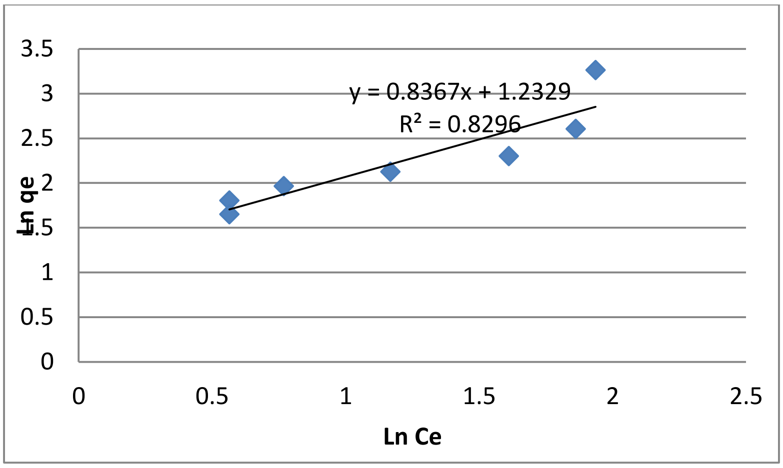


Fig: 7(Freundlich equation for chitosan Fe_3O_4 nanoparticles-Adsorbent dosage)

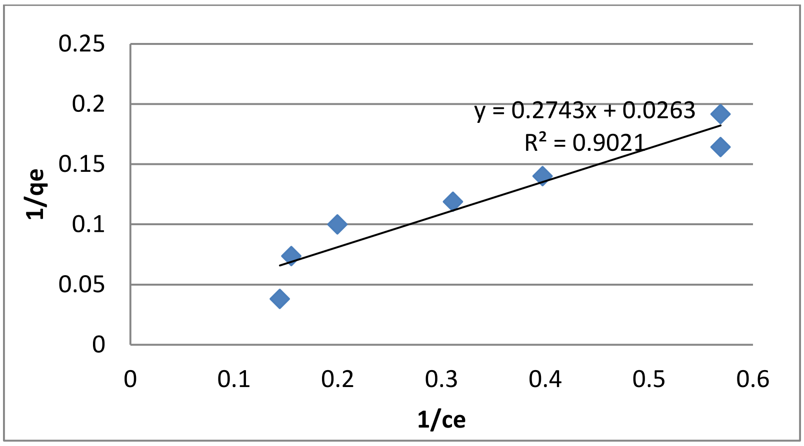


Fig: 8(Langmuir equation for chitosan Fe_3O_4 nanoparticles-Adsorbent dosage)

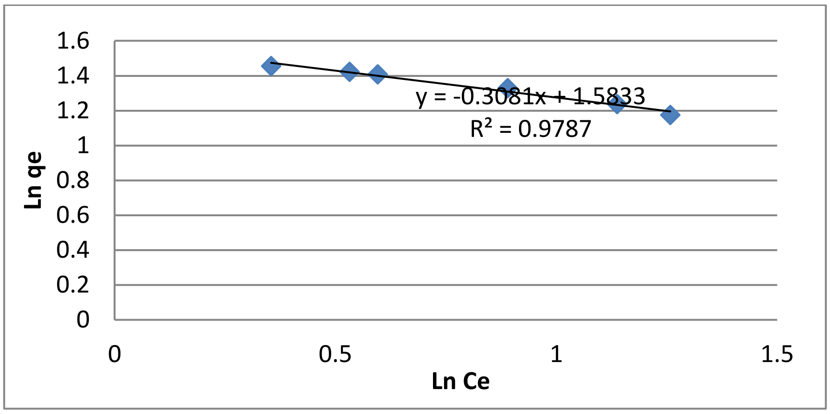


Fig:9 (Freundlich equation for chitosan Fe_3O_4 nanoparticles-PH)

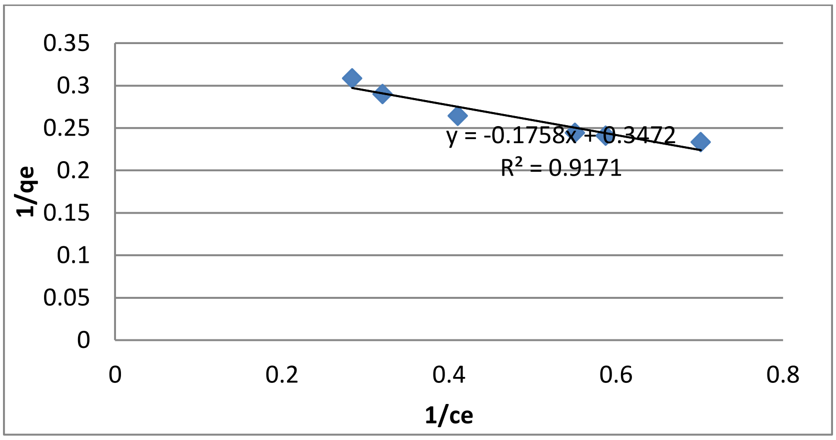


Fig: 10(Langmuir equation for chitosan Fe_3O_4 nanoparticles-PH)

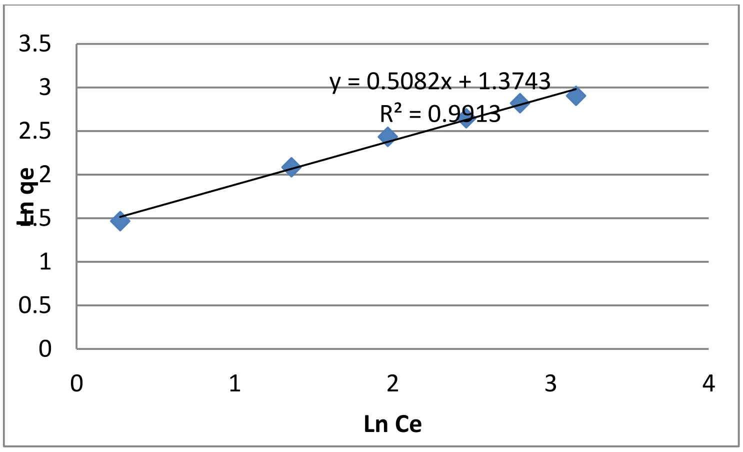


Fig:11 (Freundlich equation for chitosan Fe_3O_4 nanoparticles-Initial concentration)

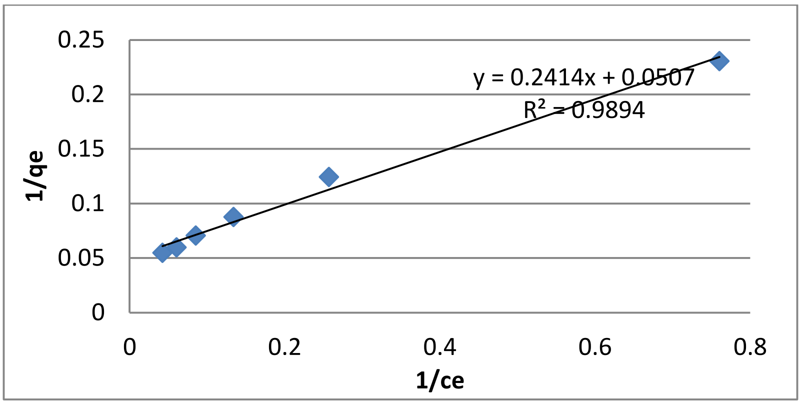


Fig: 12(Langmuir equation for chitosan Fe_3O_4 nanoparticles-Initial concentration)

Conclusion

The present study is brought out to show the suitability of the adsorbent chitosan –Ironoxide Nanoparticles for the removal of heavymetal Lead from the wastewater..Influence of process parameters such as P^H,Adsorbent dosage and initial metal ion concentration were at moderate levels such that they can affect the removal efficiencies of the heavy metals .The optimum P^H of solution for lead removal was found to be 6.Results showed that the maximum adsorbent capacity of adsorbent was at the dosage of 3g and initial metal ion concentration 10 mg/l.TGA indicate the loss of weight due to residue to moisture vapourization.SEM shows the surface morphology. The presence of Pb in EDAX indicate the adsoption of lead on the chitosan iron oxide nanoparticles .

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