Biopolymer Based Nanomaterials as Potential Biosorbents for Lead Ions

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ABSTRACT

The objectives of the present work included synthesis of nanoparticles of calcium crosslinked alginate by emulsion crosslinking method and their characterization by techniques such as FTIR, TEM and XRD. In order to study the effect of various factors like adsorbent dose, initial metal ion concentration, pH and temperature on the removal of lead ions from water using nanoparticles of alginate, a series of batch sorption experiments were conducted. The obtained results were analyzed by Langmuir and Freundlich isotherm models and it was found that the adsorption of lead more precisely follows Freundlich adsorption isotherm. The results were also analyzed through various kinetic models like Lagergreen pseudo-first order kinetics, the pseudo-second order kinetics, and intraparticle diffusion model. Adsorption of Pb ions from aqueous solutions was found to be best described by pseudo second order Lagergreen equation. It was also found that intra particle transport was not the rate limiting step. The removal of Pb ions was found to be more than 90 percent. The metal ion removal capacity of the nanoparticles was found to depend on the chemical composition of the adsorbent, concentration of the metal ion solution, pH and temperature of the medium and speed of the suspension.

Keywords: lead, alginate, nanoparticles, adsorption, emulsion cross linking**.**

INTRODUCTION

Clean water (i.e. water free of toxic chemicals and pathogens) is essential to human health. Clean water is also a critical feedstock in a variety of key industries including electronics, pharmaceuticals, metallurgical and food. The world is facing formidable challenges in meeting rising demand of clean water as the available supplies of fresh water are decreasing due to extended droughts, population growth, competing demands from a variety of users, etc[1]**.**

The current pattern of industrial activity alters the natural flow of materials and introduces novel chemicals into the environment[2]. The pollution of an aquatic environment can alter its chemical, physical and biological characteristics, jeopardizing the quality of water for human consumption[3].

Water contamination with heavy metals is a very important problem in the current world[4]. Metal ions are non biodegradable in nature and their intake at a certain level is toxic[5]. It is well known that heavy metal ions which are toxic to most organisms have found their way onto the water– system from different processes[6]. According to the World Health Organization, the metals of most immediate concern are cadmium, chromium, cobalt, copper, lead, nickel, mercury, zinc and arsenic. Some metals such as silver, mercury, cadmium and copper are markedly more toxic even at very low level. Hg, Cr and Pb are called the "big three" highly toxic. Cr, Cu, Zn are neurotoxins[7]. The most severe form of toxicity of Cd in human is "itai–itai" disease and also includes kidney dysfunction and hypertension[8]. Vapors from Hg are very poisonous, which attack the kidney and brain.

The removal of heavy and toxic metals from the aquatic environmental is one of the important issues in many industrialized countries[9]. Conventional methods for removing dissolved heavy metal ions from waste water include chemical precipitation, chemical oxidation and reduction, ion exchange, filtration, electrochemical treatment and evaporative recovery. These techniques have significant disadvantages including incomplete metal–removal. They need expensive equipments and monitoring systems, high reagent or energy requirements or generation of toxic sludge or other waste products that require disposal[10].

The selection of an effective and economic adsorbent in removal of toxic metal ion requires consideration of both conventional and non–conventional materials such as activated carbon[11], metal oxide[12], cotton[13],   jute

fibers[14], bamboo pulp[15], etc. In the last few years, biopolymers have attracted scientific community for a number of reasons. They are widely available, environmentally viable and capable of lowering transition metal ion concentration to parts per billion (ppb) concentration[16]. Another attractive feature of these biopolymers is that they possess a number of functional groups such as hydroxyls and amines to which metal ion can bind either by physical or by chemical adsorption. A more significant aspect is the metal ion recovery that may be done by simply exposing the material to aqueous acid solution.   Lead contamination of the environment is primarily due to anthropogenic activities making it the most ubiquitous   toxic metal in the environment [, 19, 20]. Research on Pb has become a   dominant topic for environmental and medical scientists for two obvious   reasons: [17] It has no known biological use and [18] It is toxic to most living things [19].

EXPERIENTAL
Materials

Biopolymer sodium alginate (Loba Chemie, Mumbai, India) was of high purity grade and purchased from local chemical supplier .Calcium chloride (Loba Chemie, Mumbai, India) was used as crosslinking agent. Silicon oil (Aldrich, USA) viscosity 10,000(At 25 ^{0} C) was used as oil phase for preparing the suspension. Lead and other chemicals were of high purity grade and throughout work, double distilled water was used for making solutions.

Methods
Preparation of Nanoparticles

The biopolymer nanoparticles were prepared following an emulsion crosslinking method. In a typical experiment, a known solution of sodium alginate were stirred on magnetic stirrer for about 20 min. 3 mL mixture of calcium chloride and silicon oil was added dropwise into the suspension of sodium alginate with the help of a syringe under constant stirring. Prepared nanoparticles were washed with acetone, then benzene and dried at 60°C for 72 h.

Characterization

FTIR Spectral Analysis

The presence of functional groups of the constituent biopolymer in the nanoparticles was confirmed by FTIR spectral analysis (Shimadzu, 1800).

Scanning Electron Micrograph (SEM)

In order to study morphology of the prepared nanospheres, the SEM analysis of the microspheres surface was carried out (STEREO SCAN, 430, Leica SEM, USA).

Particle Size Analysis

The size of the prepared nanospheres was determined by a particle size analyzer (Fritsch Particle Sizer, Germany).

Surface Charge Analysis

As the nanospheres are composed of polyelectrolyte biomacromolecular, their surface bears a change which may be determined by EMF measurements. In order to determine the magnitude of surface charge, 1 g nanospheres are allowed to swell in 25 ml of distilled water and the chare developed over the surface was measured by pH/EMF meter (Systronics   pH system 362, Ahmadabad, India). In a similar way, the Pb^{2+} adsorbed nanospheres were also immersed into same volume of water and surface charge was determined.

Adsorption Experiments

The adsorption was carried out by the batch contact method as described elsewhere[21] . Firstly, the stock solution was prepared by dissolving 0.6 g of lead (ll) acetate in 1 liter distilled water. In to 6 mL of diluted stock solution (1:1), 100 mg of swollen nanospheres were added as adsorbent. The suspension was shaken in a rotary shaker (Tempstar, India) for 4 h which was found to be a sufficient time to attain equilibrium adsorption. After shaking is over, 3 mL supernatant was mixed with 1 mL reagent (NH$_3$ solution) and the amount of Lead was assayed spetrophotometrically. The adsorbed Pb (II) was calculated by the following mass balance equation,

$$\text{Adsorbed amount (mg/g)} = \frac{(C_i - C_f) \, V}{m} \quad \dots (1)$$

where C_i and C_f being the initial and final concentrations of Lead solution (mg/mL). V being the volume of adsorbed solution and m is the weight of swollen nanospheres (adsorbent).

RESULTS AND DISCUSSION
FTIR Spectra

The Fourier transform infrared (FTIR) spectra of the sodium alginate and calcium alginate nanoparticles were recorded and compared **(Figure 1).** Spectrum of sodium alginate **(Figure 1(a))** showed important absorption bands regarding hydroxyl, ether and carboxylic functional groups. Stretching vibrations of O–H bonds of alginate appeared in the range of 3000–3600 cm−1. Stretching vibrations of aliphatic C–H were observed at 2920–2850 cm−1.Observed bands in 1649 and 1460 cm−1 were attributed to asymmetric and symmetric
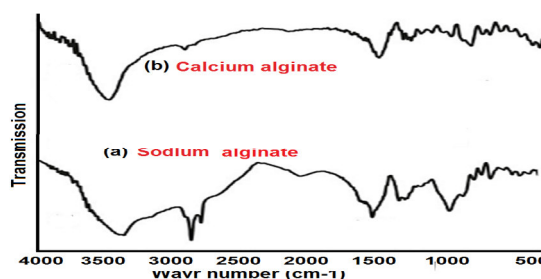

Fig: 1. FTIR   spectra of (a) calcium crosslinked alginate (nanoparticles) and (b) native sodium alginate.

stretching vibrations of carboxylate salt ion, respectively. Later bands are very significant and can be used for characterization of alginate structure from its derivatives and ingredients. The bands at 1107 and 935 cm−1 were attributed to the C–O stretching vibration of pyranosyl ring and the C–O stretching with contributions from C–C–H and C–O–H deformation Calcium alginate nanoparticles powder showed significant differences bands in comparison with IR spectrum of SA **(Figure 1(b)).** Absorption region of stretching vibrations of O–H bonds in calcium alginate appeared narrower than SA. This difference arises from the participation of hydroxyl and carboxylate groups of alginate to the calcium ion in order to form chelating structure and consequent decrease in hydrogen bonding between hydroxyl functional groups which affords narrower band in calcium alginate. Asymmetric stretching vibration of carboxylate ion shifted to lower wave numbers because when calcium metal ions replaced sodium ions in the sodium alginate, the charge density, the radius and the atomic weight of the cations were changed and hence, this shifting should be expected. It is obvious that the bands concerning carboxylate groups can be used as useful bands to follow the changes in the structure of different polymers of the alginate.

SEM Analysis

The morphology of the prepared alginate nanoparticles have been investigated by recording their scanning electron micrograph as shown in **Fig. 2**. The obtained image clearly reveals that the particles have smooth and regular morphology which is evident from the spherical shape of the nanoparticles. It also reveals from the image that the size of the nanoparticles varies between 50 to 350 nm.
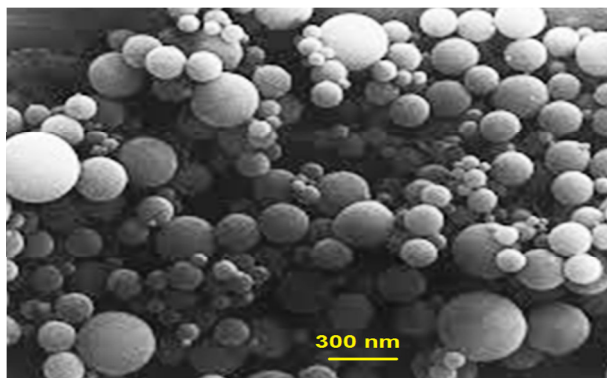
Fig:2. Scanning electron micrograph (SEM) of calcium crosslinked alginate nanoparticles.

Particle Size Analysis

The particle size analysis of the prepared nanospheres was determined by Dynamic Light Scattering measurements and the size distribution curve is shown in **Fig. 3.** It is clear from the curve that the nanospheres are heterogeneous in their average size and vary between 30 to 300 nm. It is also revealed by the Figure that majority of the particles have dimension of about 100 nm.
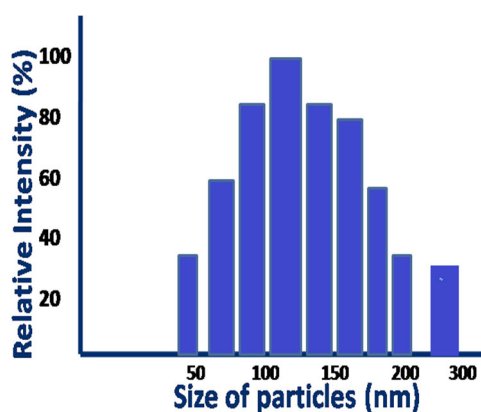

Fig. 3: Particle size distribution curve of calcium alginate nanoparticles

Results on Adsorption Studies

    (i)   Effect of metal ion concentration

The effect of initial concentration of lead ions (mg/L) on the adsorbed amount has been investigated by varying the initial concentration of Pb ions in the range 25 to 800 mg/L and determining the amount of adsorbed lead ions (mg/g). The results are summarized in **Fig. 5** which clearly reveals that the amount of adsorbed metal ions increases with increasing metal ion concentration. The results are quite expected and may be explained by the fact that with increasing concentration of metal ions, the number of lead ions approaching the solution-alginate nanoparticles interface increases which results in greater adsorption. It is worthy to note that initially the rate of adsorption (slope of the curve) is large that indicates a greater
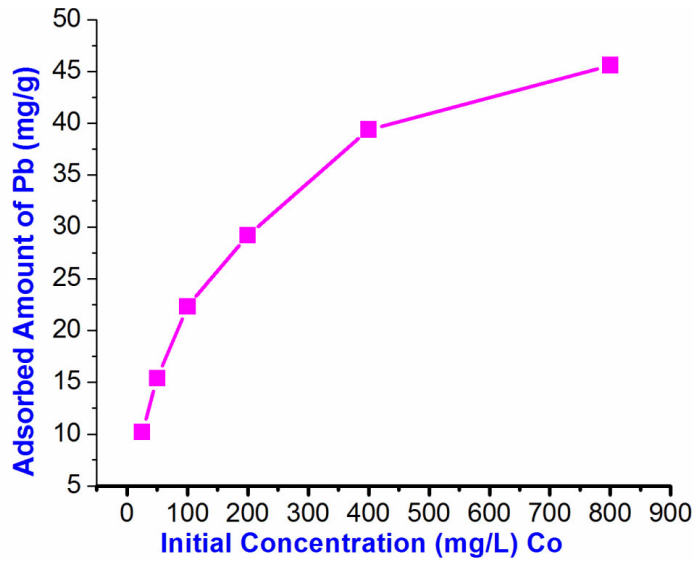
Fig. 5: Adsorption isotherm showing the effect of initial concentration of metal ions on the adsorbed amount of Pb.

intake of metal ions by the nanosorbent, however, at higher concentration of metal ions the increase in adsorption is slowed down which is evident from the observed gradually decreasing slope of the curve. Similar type of results has also been reported by other workers. **R**

Adsorption Isotherms

Langmuir Model

The effect of equilibrium concentration of metal ions on the amount of the adsorbed Pb ions may quantitatively dealt with several adsorption isotherm models among which the Langmuir equation has been the most frequently and successfully applied. According the Langmuir equation,

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \qquad \qquad \ldots\ldots\ldots(1)$$

Thus, a plot drawn between the equilibrium concentration and the adsorbed amount depicts a typical type of adsorption isotherm which is a commonly reported isotherm in the literature.
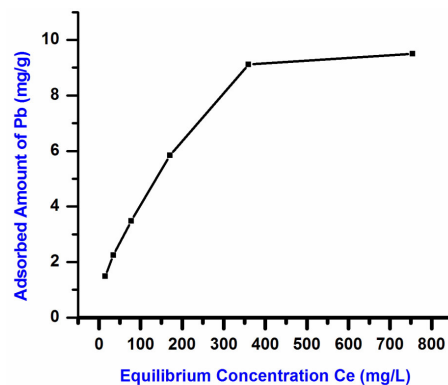

Fig. 6: A typical adsorption isotherm drawn in accordance to Eq. 1.

More popularly, the equation may also be represented by the following linear form,

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \qquad \qquad ..\ (2)$$

The linearized plot is shown in **Fig. 7** which clearly indicates that the adsorption data very well follow the Langmuir model. The values of adsorption coefficient K_L and adsorption efficiency q_m have been calculated from the eq. 2 and the values have been found to be   3   and   83   , respectively.
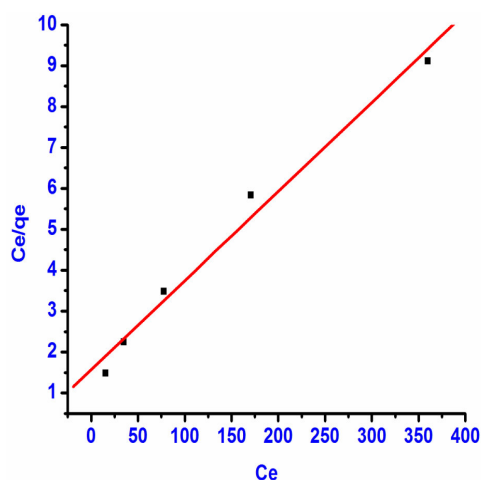

Fig. 7 A plot drawn between Ce and Ce/qe in accordance to the linearized Langmuir equation.

(ii)   Effect of pH

pH seems to be the most important parameter in the biosorption process. It affects the solution chemistry of the metal ion, the activity of the functional groups in the biomass and competition of metallic ions. Pb(ll) exhibits different type of pH dependence equilibria in aqueous solution. As pH is shifted the equilibrium will also shift. The effect of pH on the adsorbed amount of Pb(ll) ions have been investigated in the range 1.8 to 6.6 as shown in Table3.

Fig. 8 clearly reveals that the adsorption initially increases with increasing pH and becomes optimum at pH 4.0. The effect of pH on adsorption of metal ion on alginate material can be attributed to two main terms, intrinsic adsorption and Columbic interaction. The Columbic term results from the electrostatic energy of interaction between the adsorbent and adsorbate. The charge on substrates as well as softness or hardness of charge on both sides is mostly responsible for intensity of the interaction. Columbic interaction can be observed from adsorption of cationic species versus anionic species on adsorbent[22]. The results suggest that the biosorption of lead ions to the biomass is mainly due to ion attraction. Therefore, as pH decreases, the cell surface becomes more positively charged, reducing the attraction between the biomass and metal ions. In contrast, higher pH results in facilitation In contrast; higher pH results in facilitation of metal uptake. Since the cell surface is less positively charged, an optimum pH at 4.0 for adsorption of lead (II) ion was found[23].

The effect of pH on lead biosorption was examined in the pH range 1.8–6.6. At pH value higher than 6, copper ions gets precipitated due to high concentration of OH–ions in adsorption medium and subsequent studies could not be performed. At pH less than 1.8, solubility of sodium alginate is reported[24].
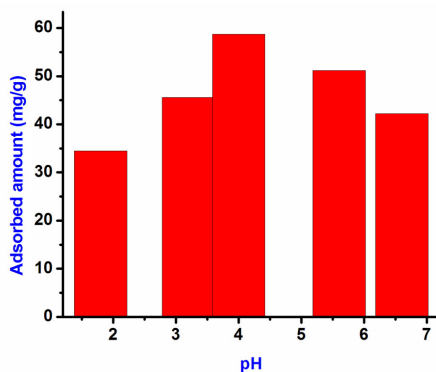
Fig. 8. Effect of pH on the adsorbed amount of Pb

(iii)   Effect of Temperature

The effect of temperature on adsorption is important not only as it affects the rate and extent of adsorption, but also due to the fact that temperature dependence of adsorption provides information about possible adsorbent–adsorbate interaction. A typical aspect of polymer adsorption is that adsorption may increase with increasing temperature, and if so, then adsorption must be entropy rather than energy favoured[25]. In the present work, the effect of temperature on adsorption was studied in the range 10 $^\circ$–40°C. The results are shown in **Fig. 9** which indicates that adsorption increases from 10 to 25°C and then starts decreasing.
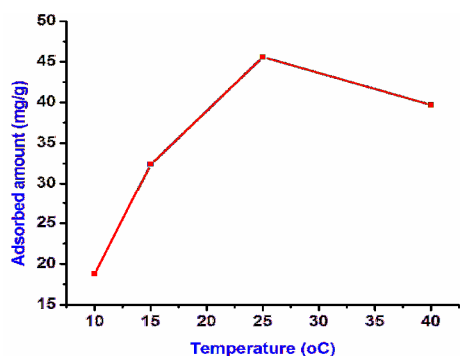

Fig. 9. Effect of temperature on the adsorbed amount of Pb

The observed results can be explained by the fact that with increasing temperature the number of active sites also increases[26]. The decrease in the adsorption of pb^{2+} ions at much higher temperature (> 25°C) could be attributed to the weakening of the binding forces between Pb^{2+} ions and active site on the biopolymer nanosphere[27].

(1)   The following thermodynamic parameters were calculated by using the following equations[28]. The standard free energy G_o (kcal/mol) was calculated by using the relation

$$G_o = -R T \ln K$$

where K being the equilibrium constant at the adsorption process. The value of   G has been calculated to be 0.949 kcal./mol

(2)   The apparent heat of reaction enthalpy H_o (k cal/mol) was estimated using equation

$$\ln k_1/k_2 = H_o/R \,(1/T_1 - 1/T_2)$$

The value of H_o has been calculated to be – 4.136 kcal./mol. The negative value of H_o indicates the exothermic nature of adsorption process.

(3)   The entropy S_o (kcal/mol) of the adsorption process was calculated using the following relationship

G_o = H_o – T S_o

The value S_o was found to be 0.0114 kcal./mol which indicate that the adsorption process is spontaneous in nature.

(iv)   Kinetics of Adsorption

A study of the rate establishment of adsorption equilibrium of polymer at the solid–liquid interface is fundamental in many areas. The progress of the adsorption process was monitored at different time intervals as shown in **Fig. 10**. It is clear from the figure that the adsorbed amount of Pb^{2+} constantly increases with increasing time and then acquires a constant value after 4 h. The rate of metal ion binding with microspheres is more at initial stage which gradually decreases and remains constant after an optimum time. At equilibrium, the removal of Pb^{2+} ions attain a constant value, because adsorption and desorption balance each other[29–30].

The observed findings may be explained by the fact that as the process of adsorption begins; the metal ions go on occupying adsorbent sites and the amount of adsorbed Pb ions increases constantly**.** However, as the adsorption process continues the number of active sites available on the adsorbent goes on decreasing which results in gradual fall in the rate of adsorption and eventually the adsorption levels off. It is worth
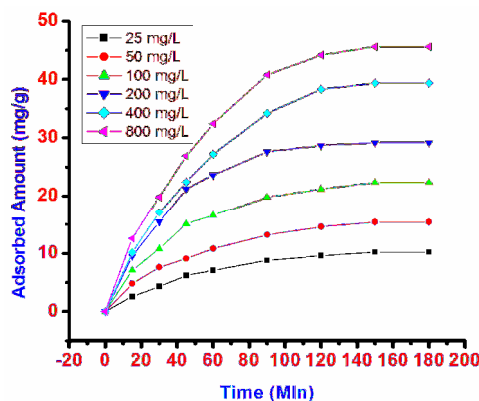

Figure 10 Curves showing the progress of adsorption process with time

to mention here that due to the reversible nature of the adsorption process the rate of adsorption and that of desorption becomes equal and a plateau is seen in the adsorption isotherm.

(v)Effect of Adsorbent Dose

The effect of the amount of adsorbent dose on the adsorption of Pb ions has been studied by adding the amount of alginate nanoparticles in the range 50 to 300 mg to the adsorption system. The results are presented in **Fig. 11** which clearly reveals that the amount of adsorbed Pb ions constantly decreases with increasing dose of the adsorbent. The observed
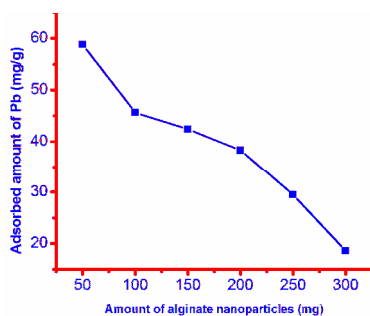
Figure 11 Effect of adsorbent dose on the adsorption of Pb ions

findings may be explained by the fact that with increasing amount of alginate nanoparticles in the metal ion solution the number of active sites also increases but at the same time the accessibility of metal ions to the active sites decreases due to crowding of adsorbent in the suspension. This obviously results in a fall in the amount of adsorbed Pb ions. Similar type of findings has also been cited frequently in the literature.

Effect of Composition of Nanoparticles

(i)   Effect of alginate

The effect of alginate content in the nanoparticles has been investigated by varying the amount of alginate in the range 1.0 to 5.0 g in the feed mixture of the nanoparticles. The results are shown in **Fig. 12** which clearly reveals that as the amount of alginate is increased
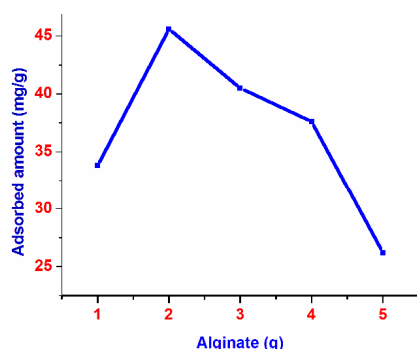

Figure 12 Effect of alginate on the adsorption of Pb ions

from 1. 0 to 3.0 g, the adsorption of Pb ions increases while beyond 3.0 g, a constant fall in the amount of adsorbed Pb is noticed. The obtained results may be explained by the fact that with increasing amount of alginate, the number of functional groups in the nanoparticles also increases while beyond 3.0 g of alginate content the nanoparticle network becomes so much compact that diffusion of Pb ions into the nanoparticle network is restrained which brings about a fall in the adsorbed amount. Similar type of results has also been reported by other workers.

(ii)   Effect of crosslinker

The number of crosslinks present in the nanoparticles has profound effect on the overall properties of the adsorbent material including adsorption capacity. In order to access the effect of concentration of crosslinker on the adsorption of Pb ions, the concentration of calcium chloride was varied in the range 0.3 to 1.0 M and the results obtained are shown in **Fig. 13**. The results clearly show that the adsorption of Pb ions increases with increasing
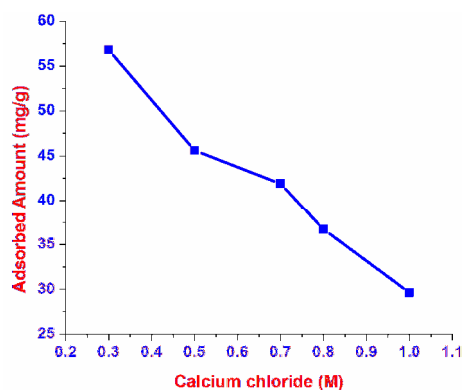
Figure 13 Effect of crosslinker on the adsorbed amount of Pb ions

concentration of crosslinker from 0.3 to 0.7 M, while thereafter a fall is noticed in the amount of adsorbed Pb ions. The results may be explained by the fact that with increasing concentration of calcium chloride the number of crosslinks also increases and due to formation of cage like structure, the lead ions more space to enter the nanoparticles network and get adsorbed at the active sites of the nanoparticles network. However, beyond 0.7 M concentration of the crosslinker, the number of crosslinks becomes so large in number that the whole network becomes much compact. This clearly results in restrained mobility of Pb ions into the nanoparticles network and as a consequence the adsorption of Pb ions decreases. Similar type of results have also been reported by other workers.

Conclusions

Chemical crosslinking of a water/oil suspension of a sodium Alginate produces regular shaped nanospheres of size up to 150 nm. The prepared nanospheres show potential for the removal of Pb^{2+} ions from aqueous solutions.

The adsorption of Pb^{2+} ions onto the water–swollen nanospheres increases with increasing initial metal ion concentration in the range 1.0 to 8.0 mg/mL. It is noticed that in the higher concentration range (8 mg/mL), the adsorbed amount nearly levels off. The adsorption process is found to follow Langmuir adsorption isotherms equation characterized by the appearance of a well marked plateau portion. The arrival of plateau is further confirmed by surface coverage–concentration plot which also exhibits more than 99 percent surface coverage in the metal ion concentration range 6 to 8 mg/mL.

The adsorbed amount of Pb^{2+} ions is significantly affected by pH of the metal ion solution when varied in the range 1.6 to 6.6. It is noticed that the adsorbed amount steeply increases in the range 1.6 to 2.4 while a marginal increase occurs beyond pH 2.4.

An optimum amount of Pb^{2+} ion is adsorbed at $25^{o}C$ while it decreases on both the sides of this temperature.

The adsorption of metal ions varies interestingly with solid to liquid ratio. When the volume of metal ion solution at constant solid is decreased, the adsorption is found to increase. On the contrary, an increase in the amount of solid (nanospheres) results in a fall in the metal ion adsorption.

The adsorption of Pb^{2+} ions also exhibits a significant change with changing concentration of the nanospheres biopolymer constituents. It is observed that there is a definite concentration of chitosan, gelatin and yeast at which the adsorption of Pb^{2+} ions is optimum and on both the sides of this optimum concentration, the adsorption is comparatively low.

The adsorption is also influenced by the chemical composition of the nanoparticles. The adsorption increases with increasing amount of alginate and calcium chloride up to a certain amount while beyond that a fall in the adsorption of lead ions is observed.

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