Studies on removal of lead ions from aqueous solutions using iron ore slimes as adsorbent

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Abstract−Iron ore slimes, a waste material generated during iron ore mining have been employed for the removal of lead ions from aqueous solutions by a batch adsorption technique. The slime sample contains 45.8% Fe, 13.6% SiO_2, and 13.9% Al_2O_3. It is characterized by X-ray diffraction (XRD) and optical microscopy to determine the presence of different phases such as hematite, goethite, limonite, quartz and kaolinite. It is assumed that the adsorption of lead ions is mainly due to the presence of pores and cavities in goethite mineral. The FTIR studies showed the presence of Si-OH and Fe-OH sites responsible for adsorption. Furthermore, the point of zero charge (pzc) of iron ore slime is shifted from 6.2 to 5.8 due to the adsorption of lead ions. Batch adsorption experiments have been conducted to study the sorption behavior of lead ions on iron ore slime. The effects of agitation time, concentration of lead ions, adsorbent doses, solution pH, other metal ions and temperature on the amount of lead ions adsorbed have been investigated. Lead ion adsorption is fast, and equilibrium could be achieved within 15 minutes of time. The adsorption increased with increase in temperature suggesting an endothermic adsorption. Under the conditions, it is possible to remove 95% lead from an aqueous solution bearing ~20 mg/*l* at pH~5.1. The equilibrium adsorption isotherm data fitted very well to both Langmuir and Freundlich adsorption models.

Key words: Iron Ore Slimes, Lead, Goethite, Mineralogy, Infra-red Spectroscopy, Point of Zero Charge, Adsorption Isotherms

INTRODUCTION

The contamination of heavy metal ions such as Pb, Cd and Hg in wastewater is a growing environmental concern as they are associated with the health risks to both human and animal lives. These metal ions are not biodegradable and tend to accumulate in the body of organisms to cause numerous diseases and disorders. Toxicological effects of heavy metal poisoning are manifested in a variety of symptoms involving high blood pressure, damage of kidney, nervous system, and reproductive system. Moreover, destruction of red blood cells, anemia, comma and death are considered amongst the most substantial consequences [1]. Out of the several toxic heavy metal ions, lead is one of the most common contaminants in water due to its wide use in paint, plastics, petroleum, ceramics, electroplating, and weapon industries. The source of human exposure to lead includes mining of complex sulfide and lead ores, smelting of lead concentrates, combustion of coal, use of lead-based pipes in water supply systems, etc. The discharge of lead ions into industrial water and effluents is of great concern because of its slow accumulation and toxic effects in living beings [2]. According to the World Health Organization (WHO), the accepted range of lead concentration in water is only 0.01 mg/*l*. Clearly, therefore, the removal of this toxic ion from the effluents is of paramount importance from the environmental pollution point of view. This needs a concerted research effort to develop some low cost adsorbents which can neutralize the harmful effect before discharge into the aqueous environment.

There are several methods to treat the toxic metal contaminated effluents such as chemical precipitation, coagulation, solvent extraction, electrolysis, membrane separation, ion-exchange, and adsorption. Most of these methods suffer from investment of high capital and cost of regeneration of materials [3]. Looking into the cost of treatment of contaminated water and effluents, many researchers have started working on different low cost adsorbents for removal of heavy metal ions from waste water. The other objective of adsorbents is also to remove higher amount of toxic metal ions in shorter time. Some of the natural adsorbents being used for the removal of heavy metal ions from aqueous systems are commercial grade natural bentonite, clay aggregate, bamboo dusts, fly ash, zeolite, activated tea waste, modified hollow fiber etc. [4-12]. Waste materials from iron and steel industries applied for the removal of lead ions from aqueous solutions [13-15] are blast furnace flue dust, Fe(III) sludge, electro furnace slag etc.

In the present investigation, iron ore slimes have been utilized for the adsorption of lead ions from aqueous solutions. Most of the iron ore mined in India generates large quantities of slimes during the process of mining and sizing of ores. These slimes are not suitable in iron and steel making due to extremely finer size and presence of higher amount of gangue constituents such as silica and alumina. Therefore, the slimes are either separately stockpiled or discarded as tailings due to a lack of suitable beneficiation techniques. Moreover, these slimes do occupy a large amount of space and cause environmental pollutions as well as ecological imbalances to the surrounding locality. Again, the production of these slimes will steadily increase with increase in production of iron ore. Several efforts are being made to enrich these vast resources of mineral waste by beneficiation techniques to earn additional revenue and conserve the mineral resources [16-18]. However, these low-grade iron materials have not yet been commercially exploited for the recovery of

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the iron values till now due to some technological constraints. Hence, as a part of our research program, for the utilization of this iron-bearing material, the main objective of this study is to determine the effectiveness in purifying the lead bearing wastewater in the surrounding mining area. Silica, alumina and iron are the three major components of iron ore slimes. Iron ore slime with high amount of goethite has a high potential as adsorbent for heavy metal ions [19]. Few researchers have experimented on certain types of iron bearing materials and found to be effective in metal ion removal [20,21] without going deep into the proper characterization of the used material. In the present study, detailed characterization studies of iron ore slime and the effect of various adsorption parameters such as time of agitation, adsorbent dose, pH and temperature have been studied on the removal of lead ions from aqueous solutions.

EXPERIMENTAL

1. Materials and Methods

The sample of iron ore slimes used as adsorbents in the experiment were obtained from an iron ore mine in Orissa, India. The sample was finely ground and used for adsorption studies. The particle size analysis of the samples was carried out by the CILAS-1064 particle size analyzer. Analytical reagent grade anhydrous lead nitrate was used in the study. A stock solution of lead was prepared by dissolving a calculated amount of lead nitrate in distlled water. Different concentrations of lead solutions were prepared by dilution of the stock solution as and when required.

Optical microscopy studies were performed to identify the mineral composition of slime samples. The X-ray diffraction pattern of the iron ore slime samples was recorded using Philips Cu-Kα radiation. The diffractograms were recorded from 10 to 70 °C with a scan speed of 30 second per step. The mineralogical studies were carried out on polished sections of samples under reflected light optical microscope. The SEM-EDS studies were done with the QEM-SCAN equipment to get an idea about the different size and shape of particles in slime sample. Zeta-potential study of the ground sample and the samples equilibrated with lead solutions were measured using the zeta probe analyzer with built-in titration system. All the samples were very finely ground, and the measurements were recorded in a dilute suspension of 1-2% solids by weight. The value of zeta potential was recorded through automatic titrations at different pH conditions. The identification of different minerals and ionic groups present in the sample was examined by the FTIR spectra (Shimadzu FTIR, IR Prestige-21) using KBr as the reference.

2. Adsorption Method

Before adsorption studies, the iron ore slime samples were thoroughly washed several times with distlled water at constant stirring to remove the soluble inorganic materials. The adsorption studies were carried out on dried sample at required pH values in 250 ml conical asks with the addition of the predetermined volume of lead stock solution. The flasks were agitated on a magnetic stirrer for the desired time period. Experiments were carried out at different parameters such as agitation time, lead ion concentration, solution pH, and temperature. The suspensions were filtered and the filtrate was analyzed for lead concentration by Perkin-Elmer, Model 2380, atomic absorption spectrophotometer (AAS). The adsorption of metal ions on slime samples was obtained by calculating the difference between the initial and final concentrations of lead in solution. The percentage removal of lead from the solution was calculated as:

Removal of lead ions (%)=(1C$_t$/C$_0$)×100,

Where, C$_o$=Initial concentration of lead in solutions (mg/l), and
C$_t$=Equilibrium concentration of lead in solutions (mg/l).

RESULTS AND DISCUSSION

1. Characterization Studies

The chemical analysis of iron ore slime was carried out by X-ray fluorescence (XRF) techniques against the known standards of similar matrix and shown in Table 1. The sample contained 45.8% Fe, 12.8% SiO$_2$, 13.0% Al$_2$O$_3$ and 8.3% LOI. The iron oxide (as Fe$_2$O$_3$) content in the sample was 81.89%. The higher value of loss on ignition (LOI) indicated (8.3%) the presence of hydroxyl-bearing minerals. The particle size analysis of the sample as used in the adsorption studies is shown in Fig. 1. It indicated that most of particles in the sample pass below 10 micron size.

2. Mineralogical Studies

The photomicrographs showing the presence of various minerals in the sample of iron ore slime are shown in Fig. 2. The microscopic studies indicated that the sample consists of hematite, goethite, limonite and quartz as the major mineral phases. Some amounts of clay minerals were also noticed within the samples. It was observed that fine grain crystals of hematite were locked within the silicates or goethites. Two types of goethites, ochreous (OG) and vitreous goethites (VG), were present in the slime sample. They

Table 1. Chemical analysis of sample

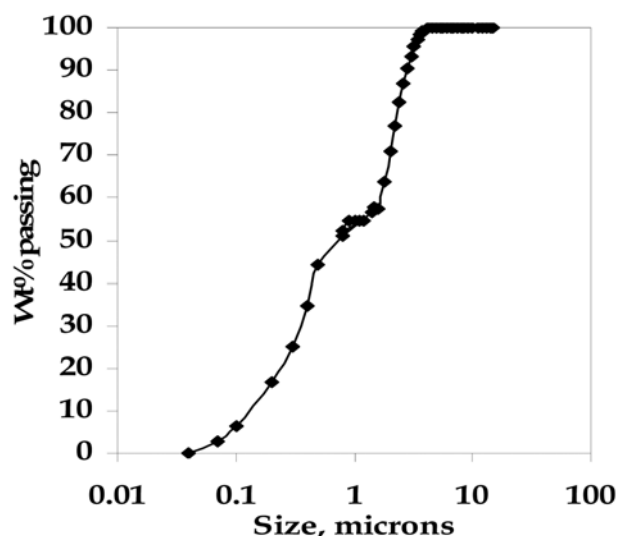| Constituents	%
Fe (T)	45.8
Fe$_2$O$_3$	65.5
SiO$_2$	12.8
Al$_2$O$_3$	13.0
LOI	8.3

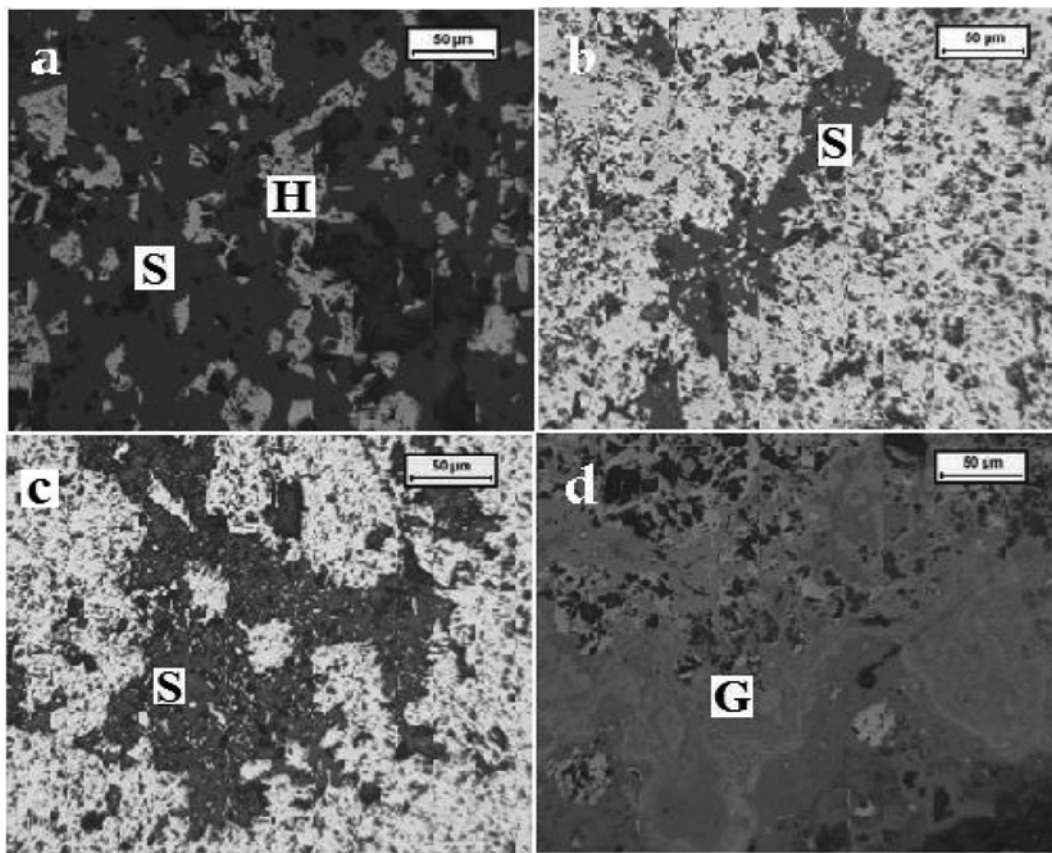

Fig. 1. Particle size analysis of slime sample.

Fig. 2. (a) Well developed euhedral crystals of hematite (H) are present as islands within the silicates (S). (b) Numerous shapes and sizes of hematite (white) are present as inclusions within the veins of silicates (S). (c) Numerous shapes and sizes of hematite (white) are present as inclusions within the clayey silicates (S). (d) Ochreous and vitreous goethites (G) are very closely and intimately present and contain numerous islands of hematite (white) as well as silicate minerals.

were very closely and intimately associated with hematite as well as clay minerals. Due to their adsorption capacity, they can incorporate variable amounts of transition and non-transition elements in the crystal structure [22]. Higher amount of ochreous goethite in bulk iron ores is responsible for the adsorption of various metal ions.

Fig. 3. X-ray diffractograms of iron ore slimes (H=hematite, G= goethite, Q=quartz).

The percentage of vitreous goethite and ochereous goethite as calculated from the modal analysis of this sample was found to be 27.44 and 25.7%, respectively.

3. XRD Studies

The X-ray diffraction patterns of iron ore sample are shown in Fig. 3. The diffraction patterns of the samples are matching well with the standard powder diffraction patterns of hematite, goethite and quartz, which are the common mineral phases associated with the iron ore samples in India. It is also known that there exist two phases, iron and silica, for adsorption of heavy metal ions.

4. SEM Study

The electron micrograph of iron ore slimes is shown in Fig. 4. The particles present in the sample are relatively fine as observed in the SEM microphotographs. The selective points, as traced for semi-quantitative analysis leading to chemical composition of the slime samples, indicate that the slime samples consist of Fe, Si, Al and O. The iron content is more in comparison to silica and alumina.

5. Measurement of Zeta-potential

The result of the zeta-potential measurement of iron ore slime in 10^{-2} N KNO_3 solutions is shown in Fig. 5. The point of zero charge was determined from the pH value at which the reversal of charge from positive to negative occurred. The results indicated that the point of zero charge of iron ore slime is at pH 6.2. It was shifted to pH 5.8 by the addition of Pb(II) ions mainly due to their adsorption
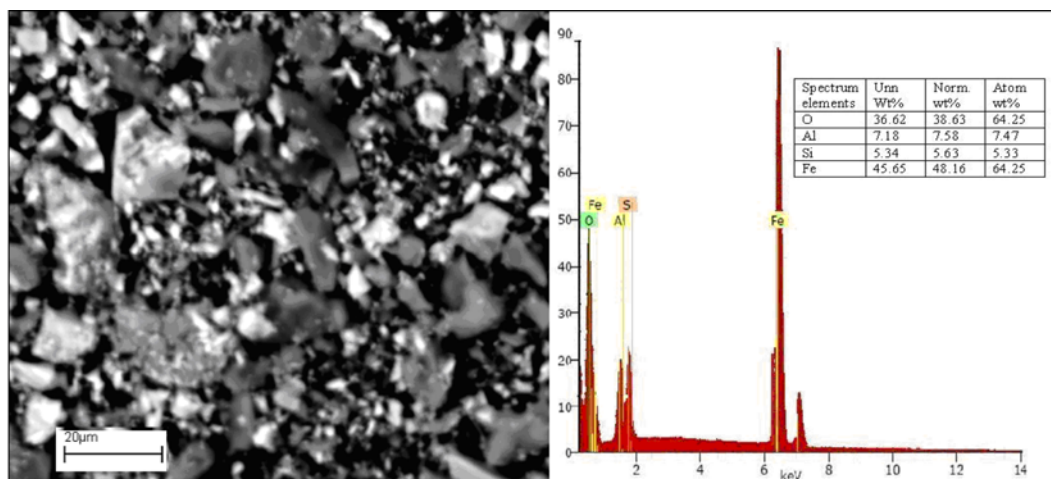
Fig. 4. SEM photomicrograph (left) with corresponding EDS analysis (right) of iron ore slime.
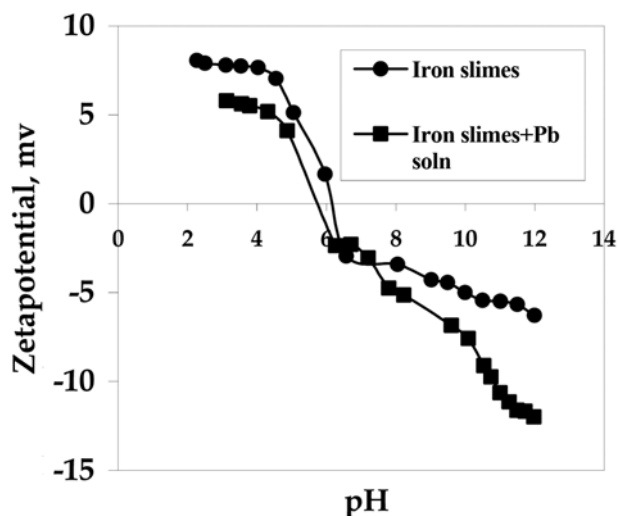

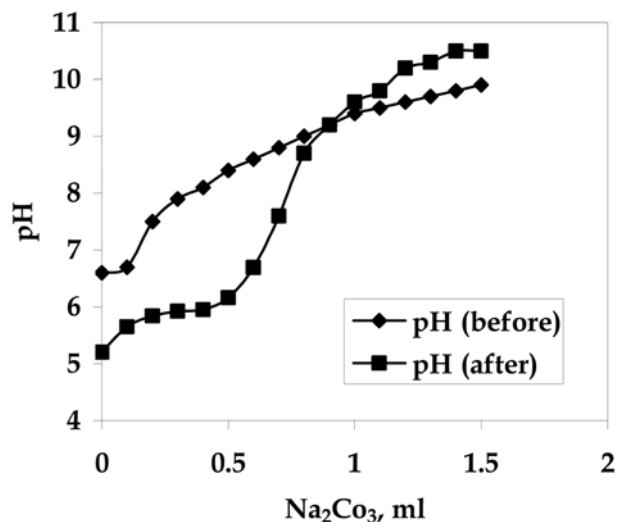Fig. 5. Zeta-potential of iron ore slimes as a function of pH.

Fig. 6. Variation of pH vs amount of sodium carbonate with iron ore slime.

by iron ore slime. The slight change of Pzc value indicates that it is due to the chemisorptions on the surface of slime samples [23,24].

The change of pH of iron ore slimes before and after absorption of Pb(II) ions was also measured with the help of pH meter by the slow addition of 0.1 N Na_2CO_3 solution. The initial pH of 1 g/l of Pb(II) solution was 6.6, and the pH of the solution decreased to 5.2 after adsorption on the slimes. It is proposed that some H^+ ions might have released during the adsorption of Pb(II) from the slime sample. The maximum pH in both the cases went up to 10.5. The change in pH plotted against the amount of sodium carbonate is shown in Fig. 6. It was observed that the adsorption of -OH ions in case of lead added sample up to pH 9.0 is slow after which it progresses rapidly.

6. FTIR Studies

Fourier transform infrared (FTIR) spectra of iron ore slime and the lead adsorbed sample are shown in Fig. 7. It is observed that the slime sample contains several mineral peaks which have been shifted due to the adsorption of lead ions. The hematite peak appearing at 500 cm^{-1} has been shifted to 479 cm^{-1}. The peak appearing
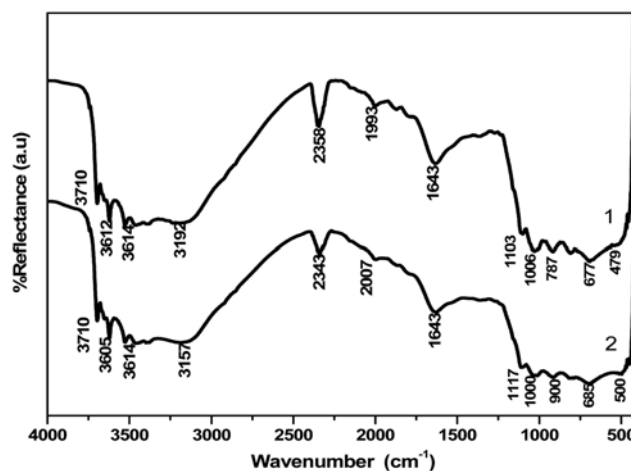

Fig. 7. FTIR spectra of iron ore slimes and Pb adsorbed sample (1. iron ore slime. 2. Iron slime sample adsorbed with lead ions).

at 696 cm⁻¹ is due to the presence of maghemite mineral appeared at 683 cm⁻¹ region. The bending vibration peak of H-O-H⁺ on the surface at 1,643 cm⁻¹ is weakened because of the adsorption of lead ions. The peak owing to Fe-OH bond is displaced from 1,026-1,103 cm⁻¹ to 1,117 cm⁻¹ with increased intensity, indicating that the inter-action is mainly through the hydrogen bond. The stretching vibra-tion peak of hydrogen bond formed between -OH on the surface of Fe_2O_3 and water molecules is displaced from 3,514 to 3,710 cm⁻¹. The spectrum showing the bands above 3,700 cm⁻¹ is assigned due to the presence of absorbed water. In the FTIR spectra, the hydroxyl stretching region of goethite causes the absorption at 3,140 cm⁻¹, and the two bands at 787 and 900 cm⁻¹ correspond to the -OH de-formation and water bending modes. The intensity of the band at 3,192 cm⁻¹ came down to 3,157 cm⁻¹ as the increase in Pb content shifted to lower energy values. The peak at 1,026 cm⁻¹ is due to (Si---O---Si) vibration mode [25,26].

7. Adsorption Studies

Various adsoption parameters for the effective removal of lead ions with iron ore slimes from aqueous solution were studied and optimised.

7-1. Effect of Agitation Time

The effect of equilibration time within 5-270 minutes on the ad-sorption behaviour of lead ions from solutions bearing 20, 50, 100, 200, 500 mg/*l* at pH 5.1 was studied. The results are shown in Fig. 8. The results indicated that the percentage adsorption of lead in-creased with increase in agitation time to reach an equilibrium con-centration. At the initial stage, the rate of adsorption of Pb(II) ions was higher, due to the availability of more active sites on the surface of slime, and it became slower at later stages due to the decreased number of active sites. It was observed that the adsorption of lead is more than 95% from solutions of lower concentration, with a small agitation time, due to the availability of sufficient vacant sites. On the other hand, the percentage adsorption decreased with the increase in initial concentration of lead ions. In all the cases, it was observed tht the equilibrium could be reached with in 15 to 20 minutes of time. However, for better adsorption of lead ions onto the iron ore sam-ple, further experiments were carried out with 60 minutes of agita-tion time.

7-2. Effect of Adsobent Doses

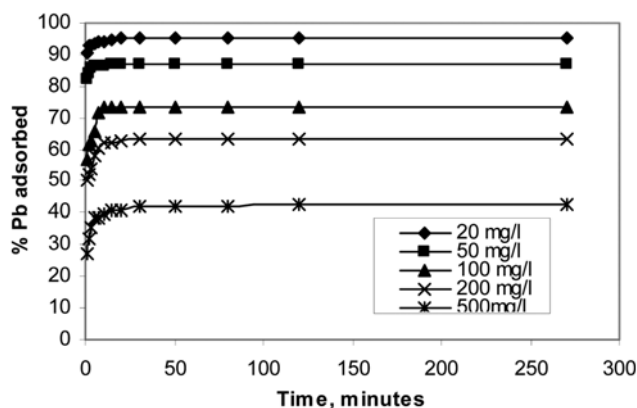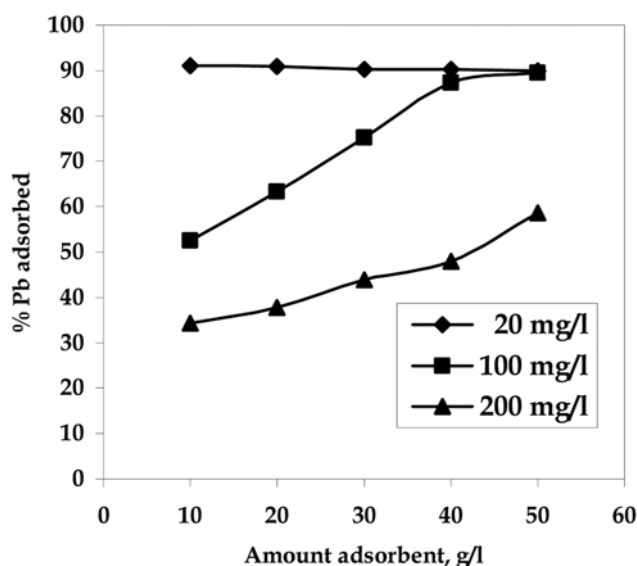Effect of adsorbent doses on the removal of lead ions are pres-

Fig. 9. Effect of amount of iron ore slimes on removal of lead ions. pH 5.1, agitation time 60 min, Temp: 300 K.

ented in Fig. 9. These studies were carried out at pH 5.1 at an agitation time of 60 minutes. The adsorption of lead increased with increase in the amount of adsorbent doses due to the availability of more surface area and active sites.

7-3. Effect of pH

The effect of pH within 2-6.5 on adsorption of different concen-trations of lead ions on the adsorbent concentration of 20 g/*l* was studied at room temperature at a agitation time of 60 minutes. The results are shown in Fig. 10. The highest rate of lead adsorption at 20 mg/*l* on iron ore slimes is more than 95% at the pH value of 3.4. At higher concentrations of Pb ions, the adsorption increased with increase in pH. It has been proposed that increasing the pH of solution leads to an increase in the negative surface charge on a sample where
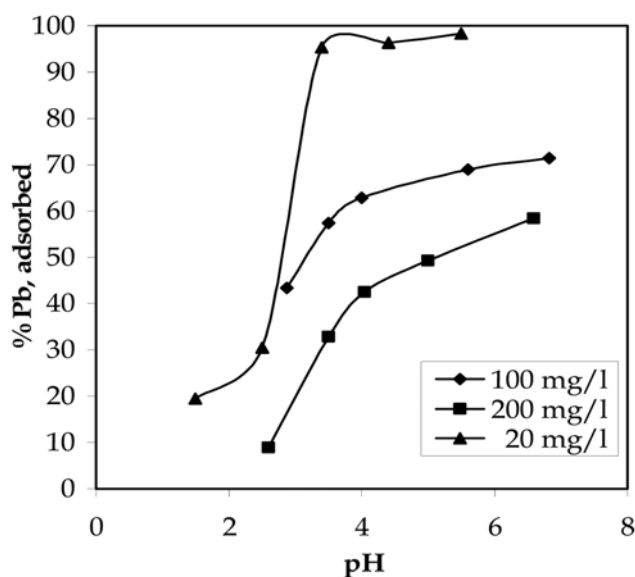
Fig. 8. Effect of agitation time on the removal of lead(II) ions by iron ore slimes. pH 5.1, adsorbent dose: 10 g/*l*, Temp: 300 K.

Fig. 10. Effect of pH on Pb(II) adsorption onto iron ore slime. Agi-tation time 60 min, Temp: 300 K.
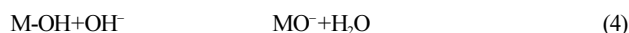
Pb^{++} ions are adsorbed by a simple binding mechanisim. Above pH 5.5, lead ions started precipitating as $Pb(OH)_2$ for which studies in alkaline range were not conducted.

7-4. Adsorption Mechanism

The speciation of Pb (II) is always pH dependent. It has been reported that at pH<6.5, lead exists in positive species while above pH 6.5 it hydrolyzed to $Pb(OH)_2$. The different species that are formed during hydrolysis are shown below [27,28].

$$Pb^{2+}+H_2O \qquad\qquad PbOH^+ + H^+ \qquad\qquad (1)$$

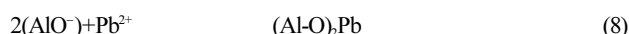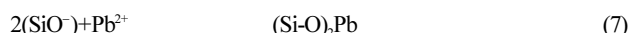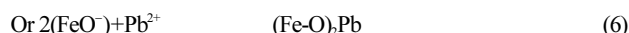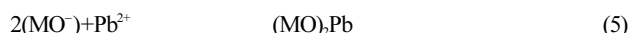$$PbOH^+ + H_2O \qquad\qquad Pb(OH)_2 + H^+ \qquad\qquad (2)$$

The surface of metal oxide of iron ore slimes also undergo protonation or deprotonation as per the following reaction.

$$M\text{-}OH + H^+ \qquad\qquad MOH_2^+ \qquad\qquad (3)$$

$$M\text{-}OH + OH^- \qquad\qquad MO^- + H_2O \qquad\qquad (4)$$

Here H^+ and OH are the potential determining ions and M is the metal ions present in iron ore slimes (Fe, Si or Al).

In iron ore slimes the affinity for Pb (II) can be described as

$$2(MO^-) + Pb^{2+} \qquad\qquad (MO)_2Pb \qquad\qquad (5)$$

$$Or\ 2(FeO^-) + Pb^{2+} \qquad\qquad (Fe\text{-}O)_2Pb \qquad\qquad (6)$$

$$2(SiO^-) + Pb^{2+} \qquad\qquad (Si\text{-}O)_2Pb \qquad\qquad (7)$$

$$2(AlO^-) + Pb^{2+} \qquad\qquad (Al\text{-}O)_2Pb \qquad\qquad (8)$$

The above equations indicate that the process of adsorption is due to columbic attraction between Pb^{2+} ions and adsorbed metal ion species. At a very low pH, two positive species will repel each other, while at higher pH significant attraction exists.

7-5. Adsorption Isotherms

Langmuir and Freundlich adsorption models were applied on the adsorption of lead ions onto iron ore slime. The Langmuir adsorption model is based on the assumption that maximum adsorption corresponds to the deposition of a monolayer of adsorbate on the surface of adsorbent. The Langmuir equation is given in Eq. (9).

$$Qe = K_L Ce/1 + a_L Ce \qquad\qquad (9)$$

The linear form of the Langmuir isotherm is given by Eq. (1):

$$Ce/Qe = 1/K_L + a_L Ce/K_L \qquad\qquad (10)$$

Where K_L (dm^3/g) and a_L (dm^3/mg) represent the Langmuir constants; K_L/a_L (mg/g) is the capacity factor of maximum adsorption (Q_{max}). Qe is the amount of solute adsorbed per unit weight of the adsorbent (mg/g) at equilibrium, and Ce is the equilibrium solute concentration in solution (mg/ dm^3).

The plot of Ce/Qe versus Ce is shown in Fig. 11. The slope and intercept represented as a_L/K_L and $1/K_L$, respectively, can be calculated from the plot. The R^2 value for the Langmuir adsorption isotherm is found to be 0.9687, which confirms the adsorption to be monolayer in nature.

The Freundlich adsorption isotherm represents the relationship between the amount of metal ion adsorbed per unit mass of the adsorbent Qe and the equilibrium concentration of the metal in solution. This isotherm is based on the assumption that the adsorption sites are distributed exponentially with respect to the heat of adsorption and is given by the equation:

$$Qe = a_f Ce^{bf} \qquad\qquad (11)$$

The linear form of the Eq. (3) is given as:

$$\ln Qe = \ln a_f + b_f \ln Ce \qquad\qquad (12)$$

Where a_f (mg/g) indicates the multilayer adsorption capacity and b_f an empirical parameter related to the intensity of adsorption. The plot of ln Qe versus ln Ce is illustrated in Fig. 12 where the slope and intercept are represented as b_f and ln a_f, respectively. The R^2 value of Freundlich adsorption isotherm is found to be 0.9839. The
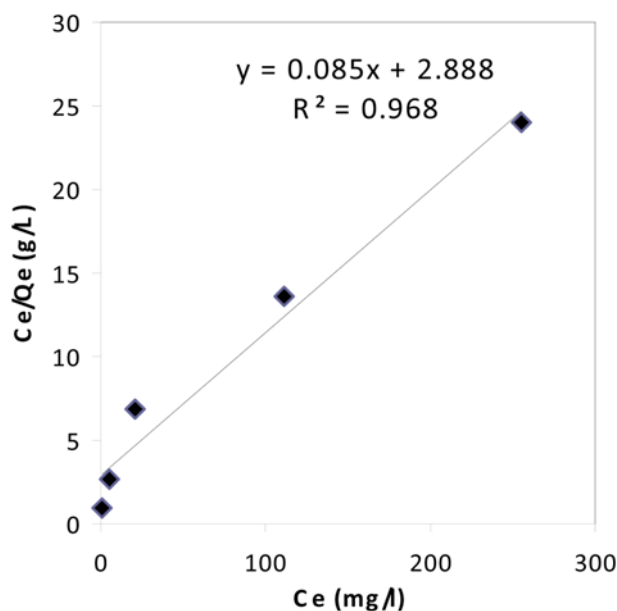

Fig. 11. Langmuir adsorption isotherms of Pb(II) ions onto iron ore slimes. Adsorbent dose: 10 g/L, Agitation time 60 min, Temp: 300 K, pH 5.1.
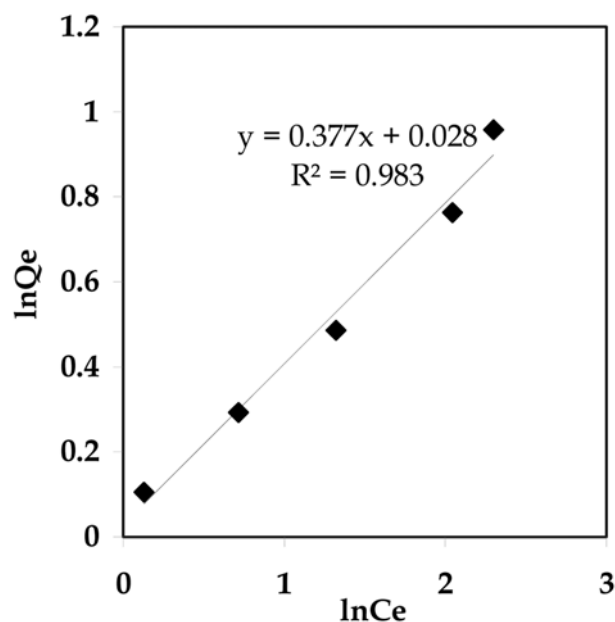

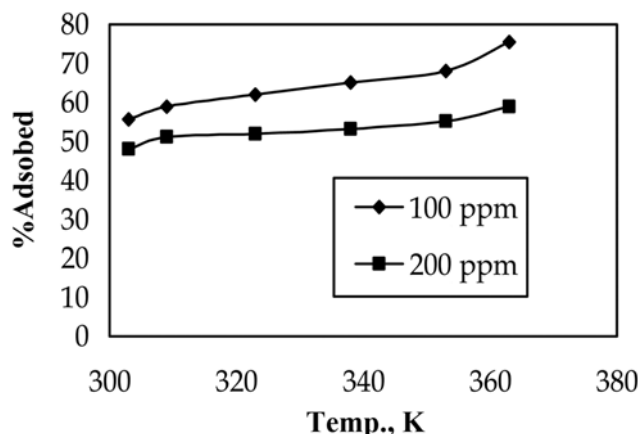Fig. 12. Freundlich adsorption isotherms of Pb(II) ions onto iron ore slimes.

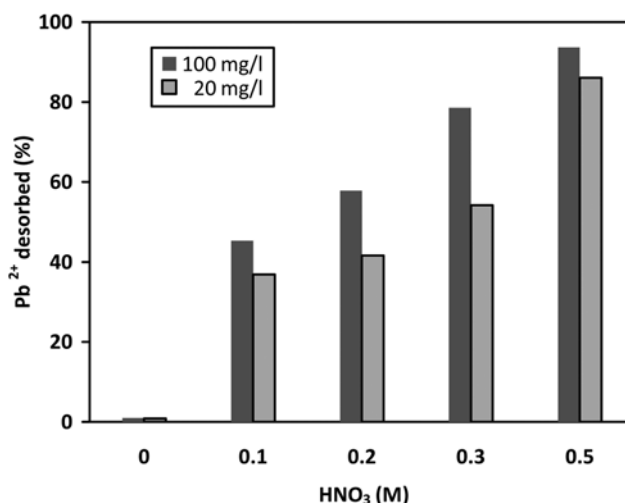Fig. 13. Effect of temperature on adsorption of lead ion (contact time 60 min, pH 5.3).

Fig. 14. Desorption of Pb(II) from lead loaded iron ore slime using different concentration of HNO₃ at two concentrations of Pb(II) solutions, adsorbent dosages 10 g/l, contact time 90 min.

results show that the adsorption of lead ions on iron ore slimes obeys both Langmuir and Freundlich adsorption isotherms, indicating the adsorption as monolayer at the beginning followed by the multilayer adsorption.

7-6. Effect of Temperature

The temperature of the system was varied from 300 to 370 K as shown in Fig. 13. The effect of temperature on the rate of adsorption indicated that the rate of adsorption increased by increasing the temperature. The increase of temperature increased the adsorption of Pb(II) ions from 55.6 to 75.46% for a 100 ppm of Pb(II) solution, indicating the process to be endothermic in nature. The increases in adsorption of Pb(II) with temperature may be due to the increase in the size of pores, and increase in intraparticular diffusion of the adsorbate, as diffusion is an endothermic process [29].

By using the following equation, the specific rate constant, K_{ad} for adsorption of lead ions on iron ore slime was determined:

$$\text{Log } (q_e - q) = \log q_e - K_{ad}t/2.303 \tag{13}$$

Where q_e is the amount of Pb(II) adsorbed at equilibrium time (mg/g), q is the amount adsorbed at time t and K_{ad} is the equilibrium adsorption constant. The plot of log $(q_e - q)$ vs. time (t) is linear and the values of K_{ad} determined at two different temperatures are 1.9×10^{-2} min⁻¹ at 25 °C and 2.1×10^{-2} min⁻¹ at 45 °C. These values clearly indicate that adsorption of metal ions is faster at higher temperatures. The activation energy for adsorption of Pb(II) ions on iron ore slimes, as determined by using the Arrhenius equation (Eq. (14)), was found to be 39.34 kJ/mol indicating strong binding sites for adsorption.

$$\text{Log} K_2/K_1 = \frac{E_a(T2-T1)}{2.303.R.T_1.T_1} \tag{14}$$

7-7. Desorption Study of Pb(II)

The desorption study of lead coated iron ore slimes was carried out by using different concentrations (0-0.5 M) of dilute HNO₃ acid solution. The iron ore slime sample loaded with lead ions was agitated for few hours with different acid solutions and than separated after filtration. The recovery of Pb(II) was calculated from the adsorption and desorption values. The results are shown in Fig. 14. Approximately 36 and 86% lead was removed by 0.1 and 0.5 M HNO₃, respectively. This desorption study suggests that lead ions

loaded with iron ore slimes can be removed by using a very low concentration of nitric acid.

7-8. Effect of Other Cations

Wastewater from any source would also contain a large amount of organic and in-organic ions that would interfere in the adsorption of lead ions. Therefore, the effect of co-existing cations such as Cd^{+2}, Cu^{+2} and Ca^{+2} on the removal efficiency of lead ions was studied. The results of the studies are shown in Fig. 15. It was observed that the metal ions have no effect on lead removal by iron ore slime. On the other hand, these ions rather adsorbed onto the slimes, suggesting the multi metal surface adsorption capacity of this material [30,31].

Taking all the above data, a 3D adsorption model with goethite (Fe₂O₃-H₂O) and Pb⁺⁺ ions is predicted based on the Avogadro software for advanced molecular editor design for molecular modeling
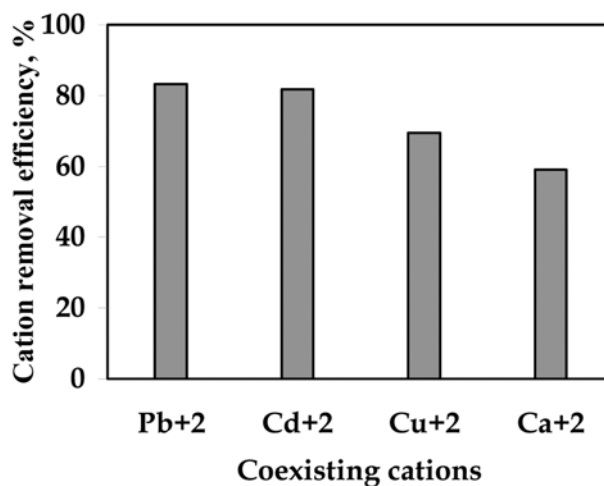

Fig. 15. Effect of coexisting cations on Pb(II) removal. Initial Pb(II) concentration 100 mg/l, adsorbent dose: 10 g/l, pH 5.1, T= 300 K.
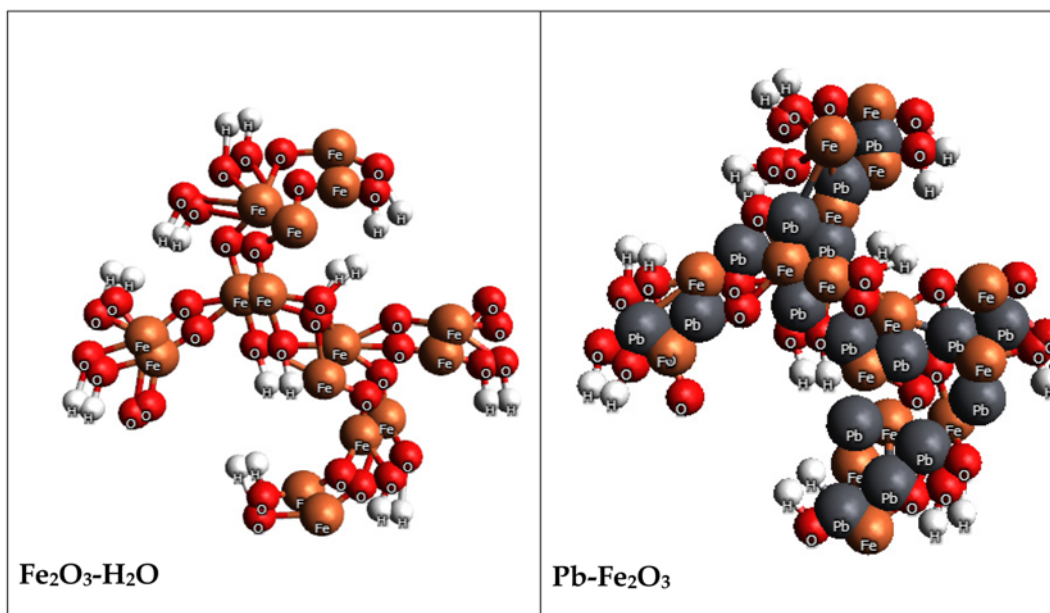
Fig. 16. Adsorption model of Pb ions on Fe$_2$O$_3$_ H$_2$O system.

as given in Fig. 16.

CONCLUSIONS

The iron ore slime sample was used as an adsorbant for the removal of lead ions from aqueous solutions. The sample was composed of several mineral phases such as hematite, goethite, clay and quartz. The adsorption of metal ions is possible due to the presence of higher amounts of ochreous goethite in the slime samples. The change in FTIR and zeta-potential values clearly indicates the adsorption of Pb ions on iron ore slimes. From the adsorption studies, it is concluded that iron ore slime is an effective adsorbent for the removal of Pb(II) ions from aqueous solutions in the concentration range of 20-500 mg/*l*. In batch studies, the adsorption was dependent on solution pH, initial Pb(II) concentration and adsorbent dose. At low concentration of Pb ions in aqueous solution, it was possible to remove around 95% of metal ion.

Adsorption of Pb(II) on iron ore slimes followed the Langmuir and Freundlich isotherm models, indicating monolayer adsorption at the beginning followed by the multilayer adsorption. The process of adsorption increased with the rise in temperature and was endothermic. The higher activation energy of 39.34 kJ/mol indicated strong binding sites for adsorption. Since the iron ore slime is a low-cost material having the advantage of economic viability, the experimental data obtained in this study may be used very well for removal of toxic metal ions from any industrial waste water or effluent.

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