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Research Article

Preparation of Nano Iron Oxide Coated Activated Sludge Granules and its Adsorption Properties for Cd (II) Ions in Aqueous Solutions

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Abstract: The purpose of this study is Preparation of nano iron oxide coated activated sludge granules and its adsorption properties for Cd (II) ions in aqueous solutions. Activated sludge as biosorbent was modified using nano iron oxide coating and examined for removal of cadmium ions from aqueous solutions. The effect of parameters including amount of coating, contact time, solution pH, initial Cd concentration and adsorbent dosage was evaluated in batch experiments. The results showed that percentage removal of 91.6 was achieved by using modified sludge at alkaline pH 9 and contact time 120 min. The uptake of cadmium was best described by Langmuir-Freundlich isotherms. The kinetic study revealed that Cd (II) adsorption was in agreement with pseudo second-order equation. The Cd (II) loaded and unloaded granules were analyzed using Scanning Electron Microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). Based on the obtained results, the modified activated sludge granules with nano iron oxide are an efficient adsorbent for removal of cadmium ions as inorganic water and wastewater pollutant.

Keywords: Adsorption isotherms, biosorbent, heavy metals, nano iron oxide

INTRODUCTION

Among heavy metals, cadmium is one of the most toxic pollutants that naturally exist in the environment. However, human activities such as mining (Meitei and Prasad, 2013) metallurgical alloying (Rao and Kashifuddin, 2012) ceramics manufacturing (Javadian *et al*., 2013) and cadmium-nickel battery manufacturing (Givianrad *et al*., 2013) are the major cause of Cd occurring in water bodies. Cadmium has been listed in EPA,s list of priority pollutants, susceptible to cause chronic health effects including cancer (Nordberg *et al*., 2005). Hence, WHO has recommended a guideline value of 0.003 mg/L for cadmium in drinking water (Sirés *et al*., 2008). There are several technologies for removal of heavy metals from aqueous solutions including chemical precipitation (Awwad and Salem, 2012; Al-Ahmary, 2009) ion exchange (Givianrad *et al*., 2013; Waseem *et al*., 2012) membrane separation (Meitei and Prasad, 2013; Awwad and Salem, 2012) electro dialysis, electro-flotation (Krika *et al*., 2011) and adsorption (Javadian *et al*., 2013; Givianrad *et al*., 2013).

One of the major techniques for removal of heavy metals from water and wastewater is adsorption. This technology has received growing attention because of advantageous such as high efficiency, simple and flexible operation (Hua *et al*., 2012; Karami, 2013) sludge-free operation and possibility of media regeneration (Zhang *et al*., 2003).

Sludge generated by wastewater treatment plants is considered as inherent adsorbent due to its porosity, high surface area and high carbon content (Xu *et al*., 2012). Using sludge as adsorbent medium is an attractive alternative for conventional disposal methods including land filling, combusting and land application. Sewage sludge and its derivates have been successfully used for removal of a wide range of pollutants such as 4-chlorophenol (Monsalvo *et al*., 2011) formaldehyde (Wen *et al*., 2011), various dyes (Li *et al*., 2011; Netpradit *et al*., 2004; Rozada *et al*., 2003) cadmium (Gutiérrez-Segura *et al*., 2012) and copper (Commenges-Bernole and Marguerie, 2009). However, as a natural adsorbent, pure sewage sludge has limited adsorption capacity that needs to be increased by the surface modification.

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Nano materials, thanks to their small size, have high surface area to volume ratio and are one of the best choices for the surface modification of adsorbents. Since, the iron based media are known as promising adsorbents for removal of heavy metals due to high affinity toward inorganic materials (Chen *et al*., 2007; Karami, 2013; Zhang *et al*., 2003) in the present study, we modified the sewage sludge with nano iron oxide particles and examined its efficacy for cadmium removal. The most important factors affecting cadmium uptake was studied at different contact time, solution pH, initial Cd (II) concentration and dose of nano-particle coated sludge. The Langmuir and Freundlich models were applied to describe the sorption behavior of the metal ions by the modified adsorbent and kinetic models (pseudo first-order and pseudo second-order) were determined.

MATERIALS AND METHODS

Preparation of sludge: Sewage sludge used in the present study was prepared from an industrial wastewater treatment plant of UASB system. The activated sludge samples were completely washed with Double Distilled Water (DDW) and then oven dried to reach to a fixed weight. After storing for 24 h at desiccators, the granules were reduced to a mesh size 10 in a standard sieves.

Preparation and characterization of nano iron oxide-granules: The treated activated sludge of 5 g was added to 10 mL solutions containing 0.25, 0.50 and 0.75 g of iron oxide nanoparticle (corresponding to 1.75, 3.50 and 5.25 w/v, respectively). A magnetic stirrer provided mixing of the solution in the vessel. After 1 h mixing, the modified sludge was washed with DDW to remove unattached oxide until the pH of the runoff water was constant (pH 7-8). The solids were dried at 105°C for 24 h and finally stored in a capped. The surface morphology of coated granules before and after adsorption experiments was observed using a-Scanning Electron Microscope (SEM) machine (Philips XL30, Holland). The functional groups were also identified using Fourier transform infrared spectrometry (FT-IR) (Bruker VERTEX 70, Germany) in the region of 4000-400 cm^{-1} wave numbers.

Batch adsorption experiments: The experiment was conducted using non-modified nano iron oxide coated activated sludge in solutions contaminated with cadmium. The effect of the most influential parameters including dose of adsorbent (2-10 g/L), contact time (10-120 min), initial solution pH (3-9) and initial metal concentration (25-125 mg/L) was examined in batch experiments. The adsorption efficiency was calculated after determination of cadmium concentration by a flame atomic absorption spectrometer apparatus (FAAS 220, Varian, Mulgrave, Victoria, Australia) at 228.8 nm. Data obtained in runs with varying dose of adsorbent were used for isotherm evaluation and those obtained in contact time varying runs were used for kinetic study of Cd (II) uptake onto coated granular sludge. The sorption behavior of the metal ions was explained by the Langmuir and Freundlich models. Pseudo first order and pseudo second order equations were applied for the kinetic study.

RESULTS AND DISCUSSION

The study was carried out in order to evaluate the effect of crucial factors on the adsorption of Cd (II) ions onto the non-modified and nano iron oxide coated granular sludge.

Effect of non-coated sludge: A series of experiment was initially conducted to evaluate the effectiveness of non-coated biosorbent at different concentrations on cadmium removal. As can be seen in Table 1, maximum Cd (II) removal was about 35%, even when the biosorbent concentration increased to 10 g/L. Moreover, increasing biomass concentration led to the decrease of adsorption capacity. These results indicated that non-coated granulates were the poor biosorbent for cadmium ions. In addition to, it seemed that at higher concentrations, aggregation of granulates decreased active sites for adsorption and biomass utilization declined, as a result. Similar outcomes have been reported in literature for other biosorbents (Bhatti *et al*., 2009).

Effect of the amount of iron oxide coating: Data on Cd (II) removal and adsorption capacity using coated granulates prepared in solutions containing different nano-iron oxide concentrations (1.75, 3.50 and 5.25%, w/v, Fe) are also presented in Table 1. Iron oxide coated sludge at dose of 6 g/L greatly enhanced the removal efficiency when compared to percentage removal of 25.08 obtained in the presence of the same concentration of non-coated biosorbent. For the three concentrations of nano-iron oxide coating, the maximum removal efficiency was achieved at 3.50% (w/v) Fe and at higher

Table 1: Cadmium adsorption performance of non coated and coated sludge at initial Cd concentration 50 mg/L, contact time 120 min and pH 7

Sorbent	Granulate dose (g/L)	Nano-particle dose (%, w/v, Fe)	Cd removal (%)	Adsorption capacity q_e (mg/g)
Non coated sludge	2	−	9.72	2.43
	4	−	17.28	2.16
	6	−	25.08	2.09
	8	−	32.76	2.05
	10	−	35.04	1.75
Coated sludge	6	1.75	54.88	4.57
		3.50	70.48	5.87
		5.25	59.24	4.93

Table 2: Adsorption characteristics of cadmium by nano iron oxide coated sludge as a function of contact time and initial pH; Cd concentration 50 mg/L, adsorbent dose 6 g/L

Contact time (min)	pH	Cd removal (%)	Adsorption capacity q_e (mg/g)
10	7	24.64	2.05
20		30.16	2.51
40		49.94	4.16
60		67.74	5.64
120		72.50	6.04
120	3	10.37	0.86
	5	24.08	2.00
	7	72.49	6.04
	8	83.78	6.98
	9	91.60	7.63

concentrations, removal efficiency decreased. These results could be explained by considering the limited sludge surface area for precipitation of nano particles (Phuengprasop *et al*., 2011). In such circumstances, excess amounts of nano particles were washed out at the end of preparation process. As a result, concentration of 3.50% (w/v) Fe was chosen for preparing coated adsorbent at the rest of the study.

Effect of contact time: Contact time is an important factor affecting economy of the process in the field application. A set of experiments was carried out with varying agitation time ranging from 10 to 120 min and the obtained results are presented in Table 2. The maximum uptake of cadmium was observed at the end of the experiment i.e., 120 min. agitation time of 120 min was recognized as the equilibrium time, meaning that at this point amounts of ions adsorbed was equal to the amounts of ions desorbed. Based on these results, the rest of the study was conducted at a fixed 120 min agitation time.

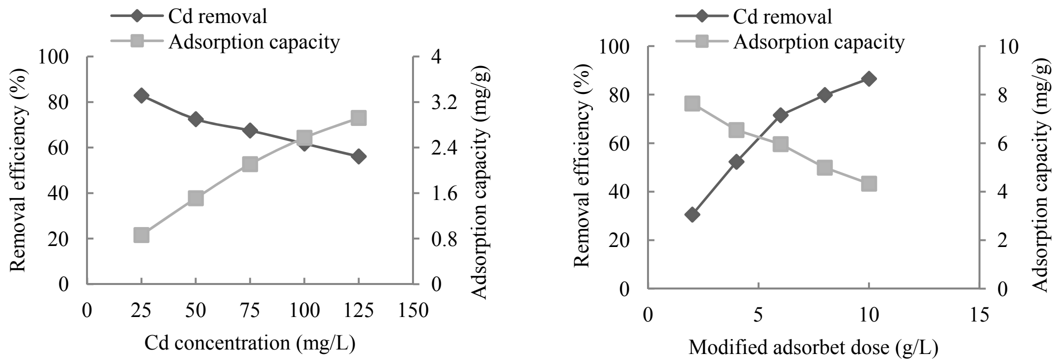
Effect of solution pH: Solution pH plays an important role in the adsorbate uptake by any adsorbent. To assess the effect of pH in a wide range of 3 to 9, the remained cadmium in the solution was measured after 120 min contact time with nano iron oxide modified granular activated sludge. The results are summarized in Table 2. As can be seen, as the initial pH of the solution increased, the removal efficiency increased. Maximum Cd removal of 91.6% was attained at pH 9 with adsorption capacity of 7.63 mg/g at equilibrium time. This effect could be attributed to the increased negative charge intensity on surface area of the adsorbent and increased affinity of the positively charged Cd (II) ions toward adsorption sites, as a result (Vitela-Rodriguez and Rangel-Mendez, 2013). Contrary, in acidic solutions a competition formed between hydrogen ion and metal ion that adversely affected the binding of cadmium on the sorption sites. Other authors also suggest similar mechanisms (Congeevaram *et al*., 2007).

Effect of initial metal concentration: Figure 1a shows the percentage removal and adsorption capacity as a function of initial Cd (II) concentration. The removal efficiency of Cd (II) decreased with increasing initial metal concentration, whereas sorption capacity increased with increasing metal concentration. This could be explained by the fact that at a fixed adsorbent dose, a fixed area of adsorption was available for Cd (II) ions. At low Cd concentrations, most of ions were adsorbed and removed from the solution, although many active sites of adsorbent remained empty. Contrary, at higher metal concentrations, the adsorbent was used more efficiently and the available adsorption sites were the limiting factor for the process. These results implied that to find out the optimal conditions for efficient removal of any adsorbate, it is necessary to make a balance between the mass of adsorbate and dose of adsorbent. The same results were also observed in other studies using sludge granules in the removal of zinc and copper ions (Bhatti *et al*., 2009).

Effect of nanoparticle coated sorbent dose: The application of nano iron oxide modified sorbent was performed by varying amount of the adsorbent. According to Fig. 1b, increasing sorbent dose up to 10 g/L led to the improved cadmium uptake due to greater available active sites of adsorbent. However, adsorption capacity was higher at lower sorbent dosage. It is also worthy to note the obtained results at the presence of coated sorbent compared to ones at the presence of non coated sorbent presented in Table 1. At the same conditions, adsorption process occurred more efficiently in nano iron oxide modified sorbent system compared to non-coated sorbent system. For example, about 87% of Cd was removed by coated sludge at 10 g/L dose; whereas the same dose of non-coated sludge could only remove approximately 35% of the metal ion. This could be attributed to the smaller pore size of nano particle coated sludge that resulted in higher surface area available for pollutant ion adsorption (Zhao *et al*., 2008). To investigate physical and chemical properties of the coated sludge sorbent, dose of 10 g/L was selected.

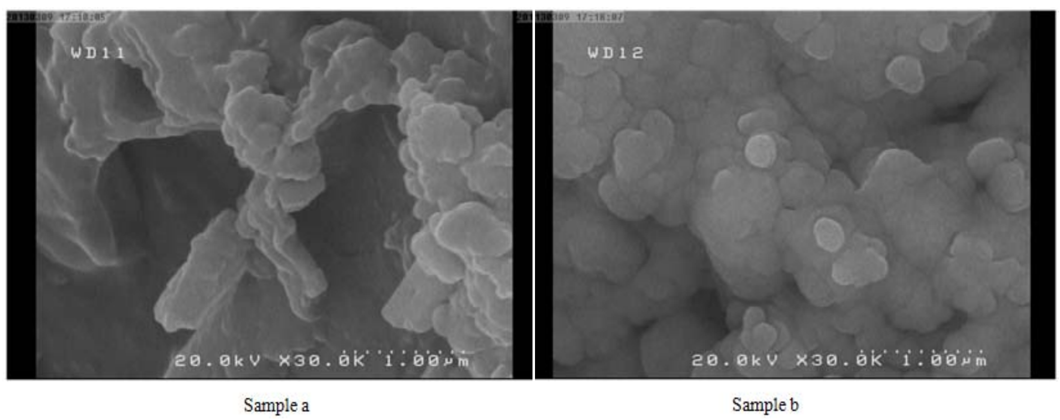
SEM analysis: The SEM analysis provided morphology of the nano iron oxide coated sludge before and after Cd adsorption. The SEM image in Fig. 2a shows porous network structure of the modified granules with a pore size of about 1 μm. The image in Fig. 2b shows a distinct morphology of granules indicating that Cd (II) was loaded on the surface of the adsorbent.

FT-IR spectral analysis: To confirm functional groups responsible for the adsorption of metal ions onto nano iron oxide coated activated sludge granules, FT-IR spectra of the sorbent was analyzed before and after Cd (II) adsorption. The most significant bands of intensity obtained from the spectrum are summarized in Table 3. The peak at 3407.52 cm^{-1} corresponding to hydroxyl group was shifted to 3287. 16 cm^{-1} after metal



(a) (b)

Fig. 1: Extent and capacity of Cd (II) adsorption as a function of a) Cd concentration and b) Modified adsorbent dose; contact time 120 min and pH 7



(a) (b)

Fig. 2: SEM images of samples, a) before adsorption and b) after adsorption

Table 3: FTIR spectroscopy bands and the possible surface functional groups of nano iron oxide coated activated sludge granules

Coated sludge (cm^{-1})	Coated Sludge+Cd (II) (cm^{-1})	Functional groups
3407.52	3287.16	−OH stretching vibration
2925.46	2920.32	−CH stretching vibration
1653.26	1656.98	C = O stretching vibration
1037.57	1035.17	C−O stretching vibration
558.81	531.86	Fe−O stretching vibration

adsorption, suggesting that Cd (II) attached on-OH group resulted in a noticeable decrease in wave number of the peak (Ahmad *et al*., 2012). The bands at around 2925, 1653 and 1037 cm^{-1} had shifted respectively to about 2920, 1656 and 1035 cm^{-1} in loaded samples compared to control. Moreover, the peak of Fe-O at 558.81 cm^{-1} was shifted to 531.86 cm^{-1} after uptake of Cd ions, indicating that iron oxide nano particles attached on granulates directly involved in the adsorption process (Yang *et al*., 2012).

Adsorption isotherms: In order to describe the Cd (II) adsorption onto the nano iron oxide coated activated sludge, the Langmuir and Freundlich adsorption isotherms were used. In Langmuir isotherm, it is assumed that monolayer adsorption occurs onto the adsorbent surface with contribution of identical sites in limited number, linearly described by Eq. (1) (Waseem *et al*., 2012):

$$\frac{C_e}{q_e} = \left(\frac{1}{q_m}\right)\left(\frac{1}{K_L}\right) + \left(\frac{C_e}{q_m}\right) \tag{1}$$

In the above equation, C_e is the equilibrium concentration of adsorbate (mg/L), q_e is the amount of adsorbate per unite weight of adsorbent (mg/g), q_m is the maximum adsorption capacity (mg/g) and K_L is the Langmuir constant. Slope and intercept of the linear C_e/q_e versus C_e plot gives the values of q_m and b parameters, respectively.

The Freundlich adsorption isotherm is a strictly empirical model describing heterogeneous systems. The linear form of Freundlich isotherm can be written as follows (Rao and Kashifuddin, 2012):

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \quad (2)$$

where, K_f and 1/n represents the Freundlich constant and the adsorption intensity, respectively. These constant values can be determined by plotting $\log q_e$ versus $\log C_e$ based on experimental data.

The linear plots for Langmuir and Freundlich isotherms are depicted in Fig. 3a and b, respectively, based on data presented in Fig. 1b. The corresponding regression coefficients, R^2, showed that both models were applicable for describing Cd (II) adsorption onto the sorbent, although Langmuir isotherm showed a slightly better fitting. The calculated Langmuir values of q_m and KL were 9.25 mg/g and 0.121 L mg^{-1}, respectively. The K_L value can be used for calculating the dimensionless constant separation factor or equilibrium parameter R_L using the Eq. (3) (Waseem *et al*., 2012):

$$R_L = \frac{1}{(1+K_L C_0)} \quad (3)$$

where, C_0 is the initial concentration of the adsorbate (mg/L). The value of R_L was found to be 0.126 for concentration of 50 mg/L of cadmium. According to literature, the R_L value in the range of 0-1 indicates favorable adsorption under experimental conditions (Waseem *et al*., 2012). It was also revealed from Fig. 3b that the Freundlich isotherm parameters of n and K_f were 2.97 and 2.31, respectively. The n value between 1 and 10 also indicated favorable adsorption (Rao and Kashifuddin, 2012).

Kinetic of sorption: Adsorption kinetics of cadmium uptake by nano particle modified adsorbent was examined using pseudo-first order and pseudo-second order kinetics models based on experimental data presented in Table 2. In the pseudo-first order equation of Lagergren, sorption capacity of solid adsorbent is used to describe the adsorption phenomena in solid-liquid interface, linearly expressed as follows (Waseem *et al*., 2012):

$$\log(q_e - q_t) = \log q_e - \frac{k}{2.303}t \quad (4)$$

In the previous equation, qt is the sorption capacity at time t (mg/g) and k is the rate constant of pseudo-first

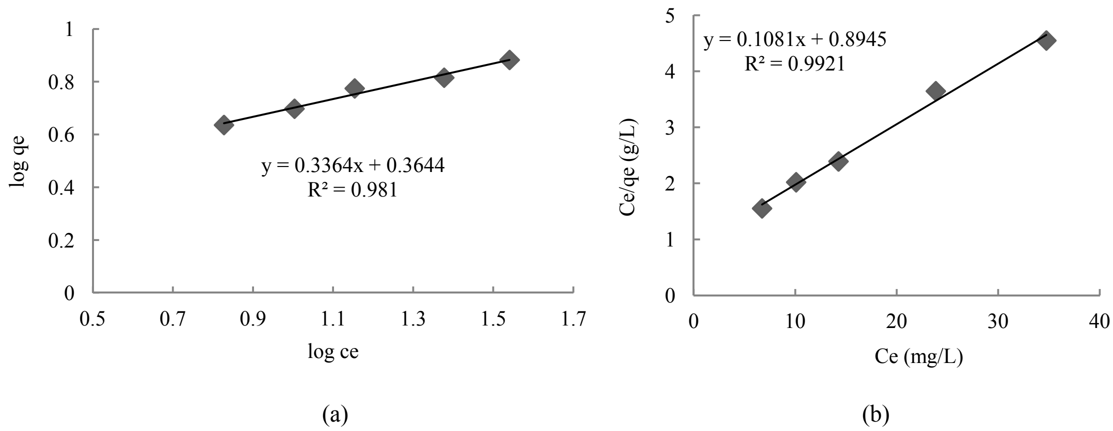


Fig. 3: Isotherm plots of Cd adsorption onto the coated granules in terms of (a) Langmuir isotherm and (b) Freundlich isotherm

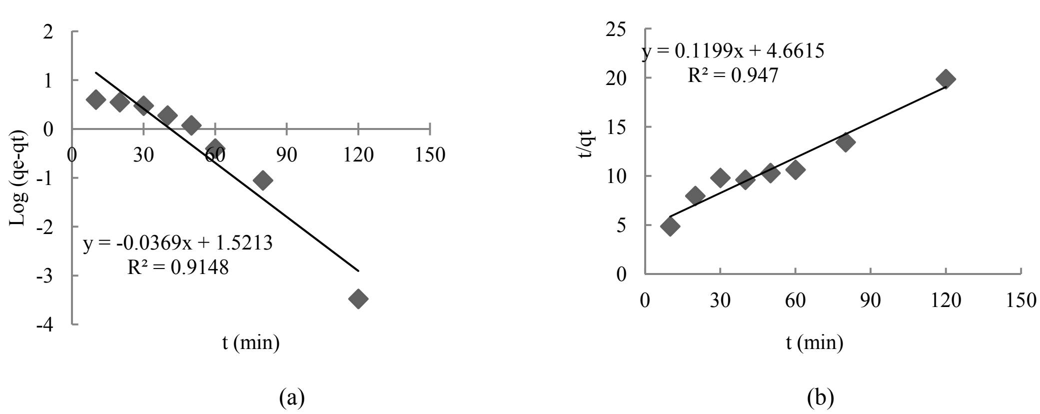


Fig. 4: Linear kinetic plots of Cd adsorption by coated granules in terms of (a) Pseudo first-order equation and (b) Pseudo second-order equation

order sorption (min^{-1}). Figure 4a depicts the plot of log (q_e-q_t) against t used for calculating q_e and k values. The calculated values were 33.21 mg/g and 0.085 min^{-1}, respectively, for q_e and k with the correlation coefficient value of 0.9148, which indicated that pseudo-first order equation was not applicable for predicting the adsorption kinetic model.

The pseudo-second order equation is used to analyze chemisorptions kinetics and can be writhen linearly in the form of Eq. (5) (Waseem *et al*., 2012):

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t \tag{5}$$

where, k is the corresponding rate constant (g/mg min^{-1}). The slope and intercept of the plot of t/q_t against t, illustrated in Fig. 4b, was used to obtain qe and k values, respectively. The calculated values were 8.34 mg/g and 3.084×10^{-3} g/mg min^{-1} respectively for qe and k with the corresponding correlation coefficient value of 0.9470. The pseudo-second order equation seemed to be the appropriate model for predicting kinetics of cadmium uptake especially when comparing the value of adsorption capacity of 8.34 mg/g, obtained by this kinetic model, with the value of 9.25 mg/g, calculated by Langmuir isotherm model. Then, it can be concluded that the adsorption was occurred due to chemisorptions (Gutiérrez-Segura *et al*., 2012).

CONCLUSION

The study was aimed to modify a natural adsorbent i.e., activated sludge from wastewater treatment plant in order to remove cadmium ions from aqueous solutions. The results showed that iron oxide nano particle coating greatly enhanced the cadmium adsorption characteristics of the sludge. Reaction time of 120 min was obtained as the equilibrium time. Increasing initial pH of the solution favored the Cd (II) uptake, whereas increasing initial Cd concentration adversely affected the removal efficiency. Maximum Cd (II) adsorption of 91.60% was achieved at pH 9 after 120 min contact time. Maximum adsorption capacity of 9.25 mg/g was calculated from Longmuir isotherm. Kinetic study indicated that adsorption of the metal ions onto the modified adsorbent followed pseudo first-order equations. The study showed that using nano iron oxide coating can efficiently modify activated sludge as a convenient and economical biosorbent.

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