

Synthesis and Application of Iron-Oxide Nano Particles Coated Sand (IONCS) For the Removal of Copper (Cu) and Chromium (Cr) from Industrial Effluent

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Abstract- Wastewater containing heavy metal ions is considered as the serious environmental problem, these have high toxic and non-biodegradable properties, which can cause severe health problems in animals and human beings. This study describes the removal of several toxic heavy metal ions i.e., Copper (Cu) and Chromium (Cr) from Industrial wastewater using Iron oxide nanoparticles coated on sand (IONCS). IONCS was prepared by the Co-precipitation method. For the purpose, locally available sand was used with prior acid activation. The IONCS sample was characterized by SEM and EDX analytical methods. The effects of different factors such as pH, contact time, amount of adsorbent, and adsorption capacity of IONCS for Cu and Cr removal were investigated. Under the optimum conditions nearly 99.88% and 100% copper and chromium removal was achieved respectively at alkaline pH condition from an industrial wastewater with initial concentration of 226.3 mg/L and 118.5 mg/L by IONCS within 105 min. The Langmuir and Freundlich adsorption isotherms were investigated. Results suggest that the Langmuir isotherm is more adequate than the Freundlich isotherm in simulating the adsorption isotherm. The adsorption capacity of the synthesized sorbent for Copper and Chromium were calculated from Langmuir adsorption isotherms in batch experiments was 3.15 mg/g ($R^2 = 1$) and 1.63 mg/g ($R^2 = 1$), and the IONCS adsorption rate constant is 29.15 L/mg and 36 L/mg respectively. The adsorption kinetic features of Cu and Cr ions were found to be fitted best by a pseudo-first-order model, suggesting the presence of physisorption processes. These findings indicate that the adsorption property of IONCS has a great potential for removal of heavy metals from wastewater.

Keywords- Copper and Chromium, Adsorption, Iron oxide, nanoparticles, sand.

I. INTRODUCTION

Huge amount of heavy metals-contaminated water are daily released to the environment worldwide as a result of

industrial activities. Human exposure to toxic heavy metals may cause many infections and diseases. Heavy metals like Copper and Chromium are major effluents from metal plating industries. They may cause mental disorder, anemia, hypertension, nausea, skin rashes respiratory problems, kidney and liver damage, lung cancer etc., in human beings [1]. So far, a number of efficient methods have been reviewed for the removal of heavy metals such as chemical precipitation, ion exchange, reverse osmosis, electro-dialysis, ultra-filtration, nano-filtration, coagulation, flocculation, floatation, etc. However, these methods have several disadvantages such as high reagent requirement, unpredictable metal ion removal, generation of toxic sludge etc. Adsorption process being very simple, economical, effective and versatile has become the most preferred methods for removal of toxic contaminants from wastewater.[2] The iron oxide nanoparticles have been utilized in various promising applications, such as catalysis, electronic devices, information storage, sensors, and drug-delivery technology, biomedicine, magnetic recording devices, and environmental remediation. Magnetic nanoparticles have large surface areas relative to their volume and can easily bind with chemicals and then they be removed using a magnet. This principal way nanotechnologies might help alleviate water problems is by removing water and wastewater contaminants including bacteria, viruses, pesticides and hazardous heavy metals like arsenic, chromium, Copper, Nickel, etc., Heavy metals contaminated wastewater from industrial activities such as electroplating, textile dyeing; tanneries etc. reach the surface or ground water sources if it is inadequately treated[3]. Iron oxide nanoparticles are gaining more attention now-a-days in environmental remediation due to their small size, large surface area and magnetic property. Iron is one of the most widespread elements in the earth. Iron oxides are chemical compounds composed of iron and oxygen. Altogether, there are sixteen known iron oxides and oxyhydroxides. Previous studies showed that iron oxide coated sand has high efficiency for removing various contaminants from water/wastewater. Application of iron oxide based nanomaterial is more attractive for removal of

heavy metals contamination from the water because of their important features like small size, high surface area, and magnetic property. Magnetic property of iron oxide nanoparticles enables easy separation of adsorbents from the system and could be reused for further application. Reusability of iron oxide based nanomaterial leads to a decrease in the economic burden. The facileness of resource and ease in synthesis render Iron oxide nanoparticles (Fe_3O_4) to be low-cost adsorbents for toxic metal sorption. Since elemental iron is environmentally friendly, Nano Iron oxide can be pumped directly to contaminated sites with negligible risks of secondary contamination. The iron oxide nanoparticles have been utilized in various promising applications, such as catalysis, electronic devices, information storage, sensors, drug-delivery technology, biomedicine, magnetic recording devices, and environmental remediation. The intensively studied Iron oxide nano particles for heavy metals removal from water/wastewater include goethite, hematite, amorphous hydrous ferric oxides, maghemite, magnetite and nano zero-valent iron. For practical applications the iron oxide nanoparticles must be coated onto some supports such as sand, bentonite, perlite etc due to agglomeration of particles, difficult separation, low hydraulic conductivity and excessive pressure drops when applied in flow through systems. One inexpensive and yet effective method is to coat sand with nano-iron oxide and use this modification to remove heavy metals. [4]

Hence, this paper focuses on the preparation of iron oxide nanoparticles coated sand by co-precipitation method for the removal of Copper (Cu) and Chromium (Cr).

II. MATERIALS AND METHODS

2.1 Reagents and instrumentation

In the present study the industrial effluent (adsorbate) from the tube valve manufacturing industry (metal plating), synthesized iron oxide nano particles coated on sand using chemical precipitation method (adsorbent) are used in this adsorption process. The chemical used to synthesis Iron oxide nanoparticles such as ferric chloride Hexahydrate and ferrous chloride Tetrahydrate were purchased from Raghu chemicals, Mysuru. Hydrochloric acid (0.1 mol L^{-1}) and sodium hydroxide (0.1 mol L^{-1}) solutions were prepared for pH adjustment. All solutions were prepared with deionized water. All bottles and glassware were acid washed and rinsed with deionized water before using. The copper and Chromium content in the wastewater samples are analyzed using an ICP – OES. The morphology and size of nano-iron oxide were surveyed on Scanning Electron – Microscopy (SEM) and Energy dispersive X-ray spectroscopy analysis was conducted

for synthesized iron oxide nanoparticles coated sand to know the elements present in the material by atomic weight percentage. Water bath shaker was used for preparation of adsorbent and batch adsorption studies.

Preparation of iron oxide nano particle coated on sand (IONCS) Sand (particle size between 0.236mm and 0.841 mm) was used as the supporting material for nano-iron oxide. Natural sand was collected and was soaked in 0.2 mol L^{-1} HNO_3 solution overnight and then rinsed 3–4 times with deionized water and dried in an oven at 100°C for 24 h. Thus, sand with acidic treated with acid was used for coating. For the preparation of IONCS, solutions of Fe (III) and Fe(II) with proper molar ratio 2:1 were prepared, respectively, by dissolving reagent grade $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ in deionized water and poured into a beaker. This beaker placed in a temperature- controlled shaker at $70\text{--}80^\circ\text{C}$ at 30 rpm and 0.1 M NaOH solution was added to it until pH reached 10. The Fe solution was mixed for another 20 min. A black precipitate was obtained confirming the synthesis of Fe_3O_4 . Subsequently, the mixed solution was poured into 100 g sand and then put in a shaker at 30 rpm for 1 h and dried in an oven at 90°C until only 10% of the water remained. The sample was then stirred at 30 rpm for 20 min to allow the stabilization of the coating process and abruptly dried at 90°C in a drying oven. The resulting sample was heated at 150°C for 8 h for complete drying and perhaps better coating stability of nano-iron on the sand surface. The small-sized particles of nano-iron oxide were aggregated on the sand surface. It was then cooled naturally to room temperature, and to remove traces of uncoated nano-iron on the sand, the dried sand was rinsed several times with deionized water, dried at 80°C and stored in a stoppered polyethylene bottle. And further this IONCS was used for the experiments [5].

Adsorption experiments

The batch adsorption study were conducted in a water bath shaker at 50rpm in 250 mL conical flask over a period of minutes to optimize the dosage and contact time. The initial Chromium and copper concentration in the industrial effluent was 117.17 and 223.74 mg L^{-1} . The adsorbents dosage was varied from 1 to 10 g/L for a period of 120 minutes, to know the effective dosage to remove Copper and Chromium which was determined using measuring the absorbance and concentration respectively. The contact time varied from 15 – 120 minutes for adsorbents with the optimum dosage. The sample were collected at regular intervals and analyzed for Cu and Cr using ICP-OES at the wavelength of 324.70 and 283 nm respectively. Optimum pH for adsorption of copper and chromium on iron oxide nano particles coated on sand was determined by varying pH 4 to 12. From the

batch studies the optimum dosage and contact time was obtained. The adsorption capacity q_t (mg/L) and percentage removal efficiency R of Copper and Chromium removal efficiency was calculated using following equation (1) and (2).

$$q_t = \frac{(C_0 - C_e)V}{m} \quad (1)$$

$$R = \frac{C_0 - C_e}{C_0} \times 100\% \quad (2)$$

Where C_0 is the initial arsenic concentration (mg L^{-1}) and C_e is the equilibrium arsenic concentration (mg L^{-1}), V is the volume of the sample solution (L), m is the weight of the adsorbent (g) and R is removal efficiency in percentage.

III RESULTS AND DISCUSSION

3.1 Adsorbent characterization

Figure 1a shows iron oxide nano particles the majority of particles have a size between 70nm – 86nm. Figure 1b shows that the surface of uncoated sand is relatively smooth and pores; The structure and morphology of the acidized sand could be beneficial to interfacial functionalized the Fe_3O_4 nanoparticles and increase further adsorption capacity of Chromium and Copper ions. However, the surface of coated sand with iron oxide nano particles shown in Figure 1c is rough. Due to deposition of iron oxide particles on the sand surface, the coated sand has more microspores and its specific surface area may be high as in comparison with uncoated sand. This is in agreement with the results of Khiadani et al. [6]. Thus, whereas acidification can penetrate gaps in the natural sand, the Fe_3O_4 nanoparticles were also shown to have successfully coated the sand.

Elemental composition of the sand was determined using EDXA spectra analysis. EDXA analysis of the unmodified sand showed the main components of the natural sand were Si – 38.93%, Al – 0.88% and O – 60.16% (Fig 2a) and synthesized Iron oxide Nano particles coated on sand (IONCS) are Si – 13.9%, Al – 0.43%, C – 8.71%, O – 47.89%, Cl – 2.07%, Na – 10.64% and Fe – 16.78% and from the SEM images the natural sand morphology was smooth surface, synthesized IONCS have the rough clustered surface. After coating of sand with nano iron oxide, the iron content of the sand was increased to 16.78 % of its elemental composition (Fig 2b). In contrast to our study, the size of coated particle in Khiadani and coworker study was not in nano size. Coating with nano particles caused the iron oxide penetrate to deeper layers of the sand and provide a larger interlayer space which may lead to larger micro pore diameters and spaces [6].

3.2 Effect of adsorbent dose

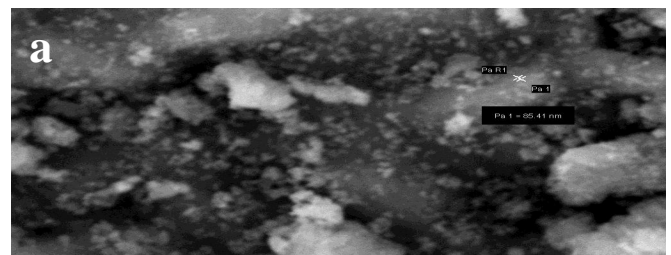
Various IONCS adsorbent doses are added to the aqueous solution to determine the maximum adsorption capacity of the IONCS for Cu and Cr with an optimum IONCS dose. The adsorbent dose was varied from 1 – 10 g/mL of wastewater. 100 mL of sample was taken in a conical flask and the adsorbent dosage was added and allowed in water bath shaker for 120 min. The concentrations of Copper and Chromium used for determination of optimum adsorbent dosage were 214.33 mg/L and 121.92 mg/L, respectively. A plot of IONCS dose against percentage removal of Cu and Cr are shown in Figures 3.

The removal efficiency of copper decreased from 16.98 % to 95.10 % with the increase in the adsorbent dosage from 1 g/L to 10 g/L. Similarly, the removal efficiency of Chromium also decreased slightly from 36.75 % to 98.58 % with the increase in the adsorbent dosage from 1 g/L to 10 g/L.

In the present study the optimum dosage was found to be 7 g/L of IONCS for an effective adsorption of Copper and Chromium. Iron oxide nano particles coated sand (IONCS) showed 97.35 % and 99.98 % for Copper and Chromium adsorption efficiency at the optimum dose.

3.3 Effect of Contact Time

The effect of contact time was studied as a function of removal of Copper and Chromium for IONCS adsorbent. To study effect of contact time, the optimum dosage of 7 g/L of adsorbent dose was added to 100 mL of wastewater respectively. The effect of contact time for removal of Copper and Chromium shown in Fig 4, from this figure it is observed that there was gradual increased in the removal of Copper and Chromium till 105 min and gradually it reached the equilibrium. This is due to the availability of the surface on the adsorbents for adsorption at the initial time and the after the equilibrium is achieved the voids of the adsorbents for which the removal of Copper and Chromium efficiency become constant. The optimum contact time attain equilibrium was found to be 105 min.



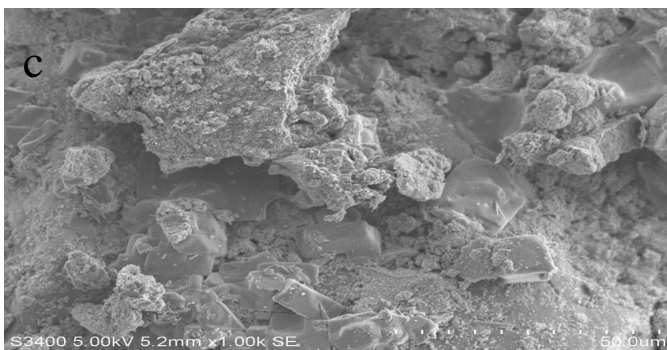
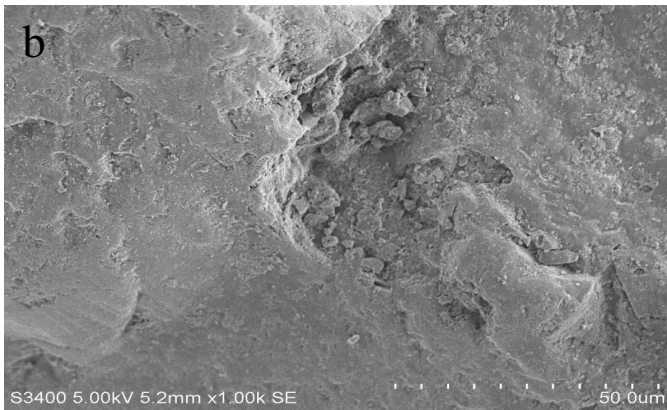


Fig 1 SEM images of (a) Iron oxide nano particles (b) Natural sand (c) Synthesized Iron oxide nano particles coated sand (IONCS)

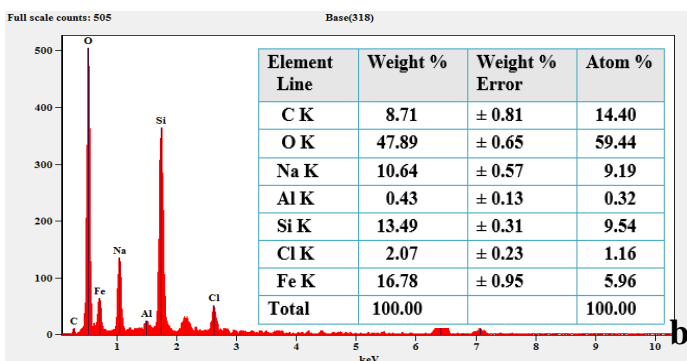
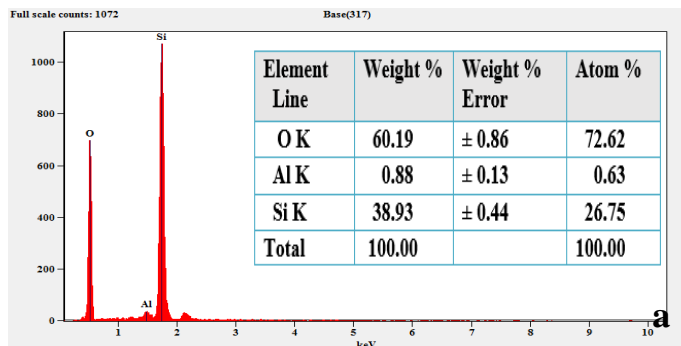


Fig 2. EDS analysis of (a) Natural sand and (b) Synthesized Iron Oxide Nanoparticles Coated Sand (IONCS).

3.4 Effect of pH

The hydrogen ion concentration in moles per liter is given by pH of the solution. pH is the negative logarithm to base 10 of the activity of the hydrogen ion ranging from 0 to 14 where the pH 7 indicates the neutral condition. In the present study, effect of pH on the Cu and Cr removal was carried at varied pH in the range between 4 and 12. The pH of the wastewater was maintained by adding HCl and NaOH. Both the heavy metals showed maximum adsorption at neutral and alkaline pH. At pH 12 and 7 Copper and Chromium adsorption efficiency was observed nearly to 100% for the adsorbent dose 7 g/L Shown in Figure 5. From these plots an optimum pH maintained for batch adsorption for copper and Chromium was found to be 7.

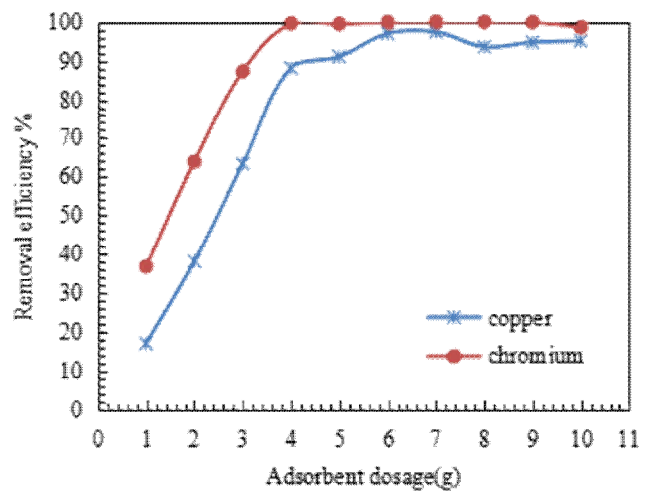


Fig 3 Effect of IONCS dose on adsorption of Copper and Chromium

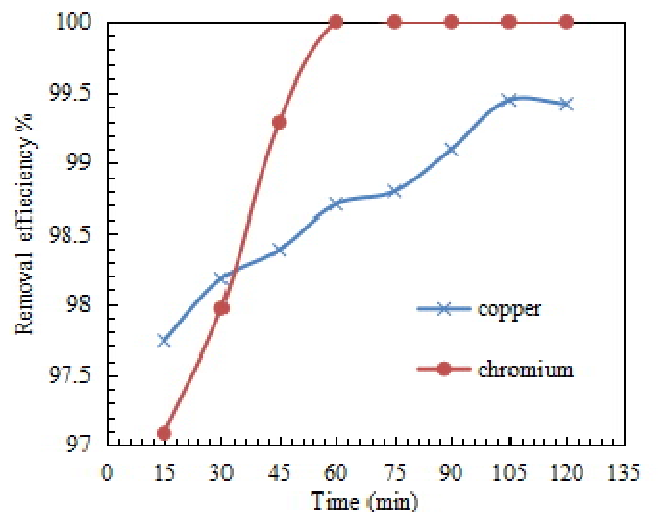


Fig 4 Effect of Contact time on adsorption of Copper and Chromium

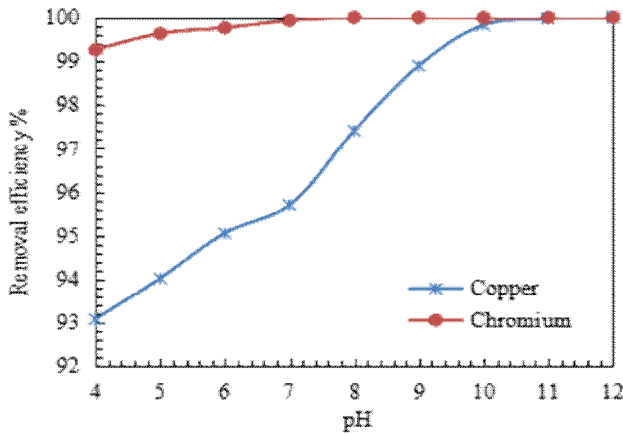
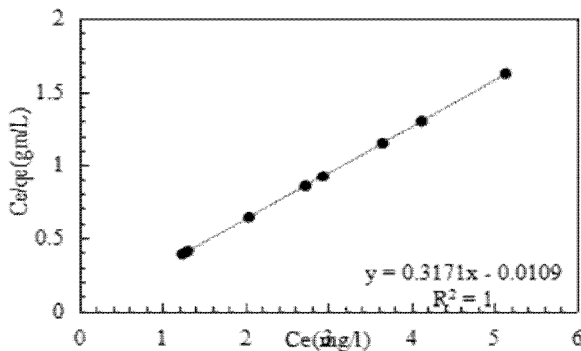


Fig 5 Effect of pH on adsorption of Copper and Chromium

Higher adsorption rates of Cu and Cr on to the IONCS was found to be at alkaline pH, which may be due to the effect of higher electrostatic repulsion that exists between the positively charged Cu and Cr ions and positively charged surface of Iron oxide nano particles coated on sand. The positively charged sites on the system decreases as the pH in the system increases, resulting in better sorption at alkaline pH.



3.5 Adsorption Isotherm Studies for Copper and Chromium

The equilibrium established between the liquid and solid phase can be represented by a quantitative relationship called adsorption isotherm. It also describes the distribution of adsorption molecules at equilibrium on the two phases. The adsorption of Cu and Cr can be optimally designed for the adsorption system by applying the correlation between adsorption mechanism and equilibrium constants. It also provides the adsorption capacity of the adsorbent also the mechanism involved in the adsorption process. The adsorption isotherms relate the amount of adsorbate adsorbed on adsorbent at the interface and the concentration of adsorbate in the solution. In the present study batch experimental data were used to check the applicability of Langmuir and Freundlich isotherms on Cu and Cr adsorption.

The linearized Langmuir and Freundlich isotherm model can be expressed by Eqs. (3) and (4), respectively:

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m} \tag{3}$$

$$\log q_e = \log KF + \frac{1}{n} \log C_e \tag{4}$$

Where C_e (mg L^{-1}) is the equilibrium concentration of adsorbate in solution, q_e (mg g^{-1}) is the equilibrium adsorption capacity, q_m (mg g^{-1}) is the maximum adsorption capacity of MIONCS for monolayer coverage, b (L mg^{-1}) is a constant related to adsorption free energy, KF ($\text{mg l}^{-1/n} \text{L}^{1/n} \text{g}^{-1}$) is a constant related to sorption capacity, and n is an empirical parameter related to sorption. Values of q_m and b can be calculated from the slope and intercept of linear plots of C_e/q_e vs. C_e for Eq. (1) and values of K_F and n can be obtained from Eq. (2).

3.5.1 Langmuir Adsorption Isotherm

Langmuir isotherm described quantitatively the formation of a monolayer adsorbate on the outer surface containing a finite number of identical sites of the adsorbent and after that no further adsorption takes place. Thereby, the Langmuir represents the equilibrium distribution of metal ions between the solid and liquid phases.

In the present study Langmuir constants for adsorption of copper and Chromium on to the IONCS was determined. A plot has been made C_e/q_e versus C_e and the value of K_L and q_m were determined for the adsorption of Cu and Cr on Iron oxide nano particles coated on sand (IONCS) [Figure 6 (a) and (b)]. The maximum adsorption capacity q_m were found to be 3.15 and 1.63 mg/g for Cu and Cr, respectively. The value of 'R²' showed that Cu and Cr had correlation value of 1. This was well supported by the maximum adsorption capacity q_m , since the values for Cu was double compared to Cr.. The values of K_L and q_m for both the adsorbates determined is presented in Table 1

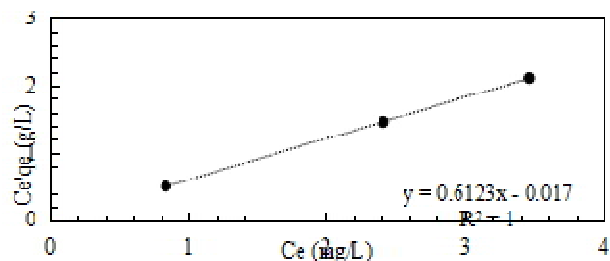


Fig. 6 Langmuir isotherm for adsorption of (a)Copper (b)Chromium on Iron Oxide Nano particles Coated on Sand (IONCS)

Table 1. Langmuir Isotherm Constant

| Heavy Metals | K_L (L/mg) | q_m (mg/g) | R^2 |
|--------------|-----------------|-----------------|-------|
| Copper | 29.15 | 3.15 | 1 |
| Chromium | 36 | 1.63 | 1 |

3.5.2 Freundlich Adsorption Isotherm

Freundlich isotherm is commonly used to describe the adsorption characteristics for the heterogeneous surface. Freundlich isotherm was applied for IONCS Cu and Cr system. Plots of log q_e versus log C_e has been made is depicted in Figure 7. From these plots the Freundlich constants such as ‘KF’ and ‘n’ were determined. The adsorption intensity or the heterogeneous surface can be obtained by slope where the value of $1/n$ ranges between 0 to 1. As the value of the $1/n$ appears near to zero the system indicates more heterogeneity. In the present study, for the adsorption of Cu and Cr present in the tube valve manufacturing industrial wastewaters, the values of $1/n$ and KF were found to be 0.0116 and 3.23 for Cu and for Cr was 0.0022 and 1.6838, respectively. From the graph (Figure 7(a) and (b)) log q_e versus log C_e it is observed that the data is best fitted with Cr and the value of R^2 is greater than Cu. The isotherm constants ‘ $1/n$ ’ values indicate that adsorption is highly favorable with the Freundlich model, since the value of ‘ $1/n$ ’ is found to be below 1. The values of Freundlich equilibrium constant KF and Freundlich constant n are represented in Table 2.

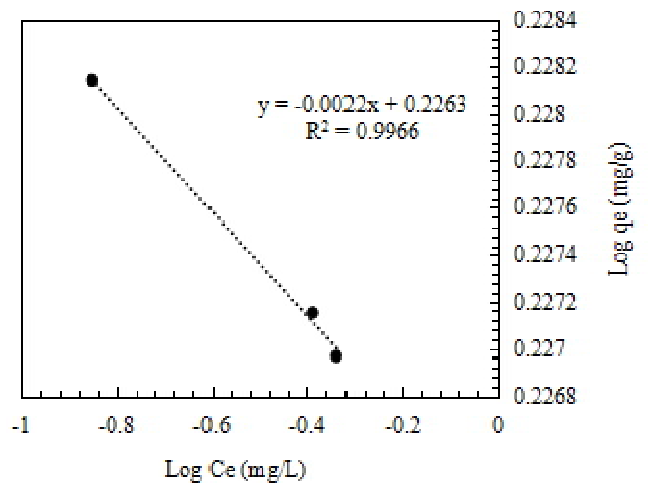
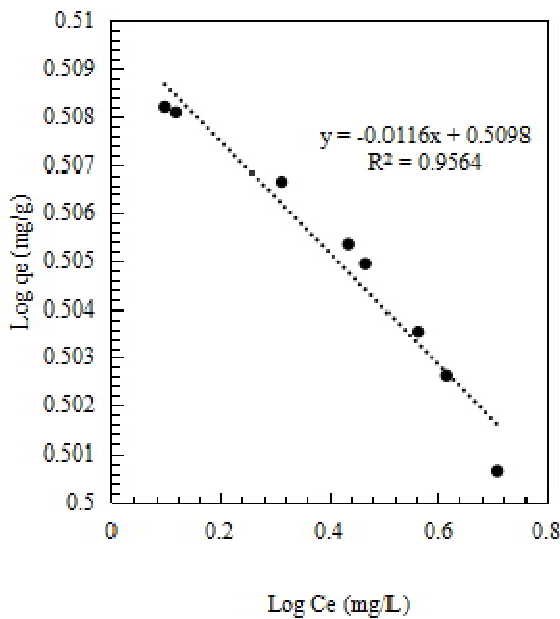


Fig.7 Freundlich isotherm for adsorption of (a) Copper (b) Chromium on Iron Oxide Nano Particles Coated on Sand

Table 2 Freundlich Isotherm Constant

| Heavy Metals | K_F (L/mg) | $1/n$ | R^2 |
|--------------|-----------------|--------|--------|
| Copper | 3.23 | 0.0116 | 0.9564 |
| Chromium | 1.6838 | 0.0022 | 0.9966 |

3.6 Adsorption Kinetics Model

For the equilibrium data obtained from the present study the adsorption kinetics pseudo first order and pseudo second order kinetics were studied in order to determine and interpret the mechanisms of metal adsorption process and the main parameters governing sorption kinetics [7].

3.6.1 Pseudo First Order Kinetics

The rate constant of adsorption is determine from the pseudo first order equation given by equation 5.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{5}$$

Where, q_e and q_t are the sorbed concentration at equilibrium (mg/g) and at time t in min. k_1 is the first order rate constant. The value k_1 has been calculated from the plots of $\ln(q_e - q_t)$ versus t shown in the Fig 8 (a) and (b). These plots represents the pseudo first order kinetics for the adsorption of Cu and Cr concentration from tube valves manufacturing industrial wastewater on the adsorbents of IONCS. The values of k_1 was found to be 0.0362 and 0.0466 for Cu and Cr respectively. The value of q_e was found to be 29.54 mg/g and 9.70 mg/g for Cu and Cr on IONCS respectively as shown in table 3.

These figures showed that the adsorption follows the pseudo First order kinetics which means physisorption.

Table 4.5 Pseudo Second Order Kinetic constants

| Metals | k_2 | q_e mg/g | R^2 |
|----------|--------|------------|--------|
| Copper | 0.0334 | 1.1388 | 0.8825 |
| Chromium | 0.143 | 0.60931 | 0.8697 |

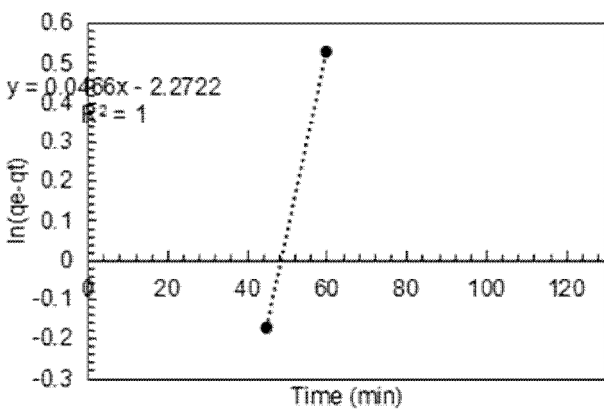
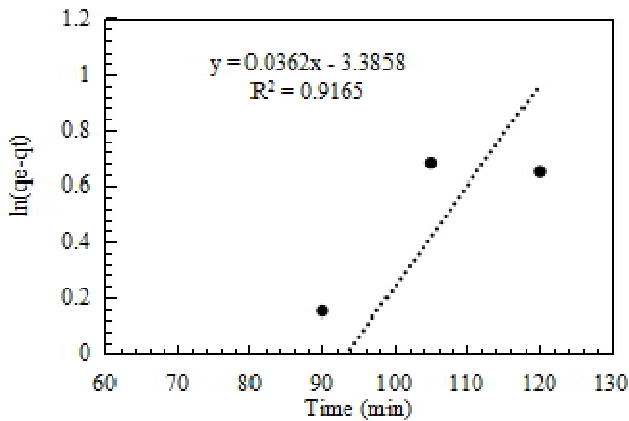


Fig 8 Pseudo first order kinetics for synthesized IONCS (a) Copper (b) Chromium

Table 3 Pseudo First Order Kinetic constants

| Metals | k_1 | q_e mg/g | R^2 |
|----------|--------|------------|--------|
| Copper | 0.0362 | 29.54 | 0.9165 |
| Chromium | 0.0466 | 9.70 | 1 |

3.6.2 Pseudo Second Order Kinetics

A pseudo second order equation is based on equilibrium adsorption is expressed in equation 6.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

Where, k_2 is the rate constant of second order adsorption g/mg/min. The value of k_2 and q_e has been evaluated from the plots of t/q_t versus time t shown in Fig 9 (a) and (b). The pseudo second order kinetics constant are given in table 4.. The value of k_2 was found to be 0.0334 and 0.143 for Cu and Cr respectively for IONCS. The value q_e was found to be 1.1388 and 0.60931 for Cu and Cr respectively.

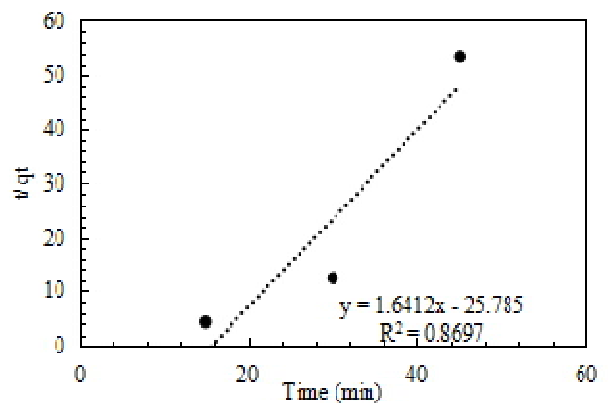
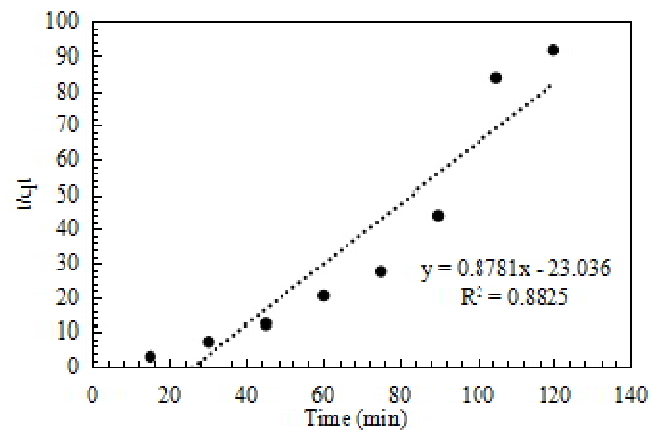


Fig 9 Pseudo Second order kinetics for synthesized IONCS (a) Copper (b) Chromium.

IV. CONCLUSION

In this study adsorbent Iron oxide nanoparticles coated sand (IONCS) were synthesized by co-precipitation method. And it was used for the removal of Chromium and Copper from industrial effluent by batch adsorption experiments. Iron oxide nanoparticles were characterized by SEM analysis the results showed that the size of nanoparticles were 80-90nm. SEM images showed the sand surface was smooth and the sand coated with Iron oxide nanoparticles were crystalline and rough. EDS analysis were determined to know the elemental composition, the Iron content on sand was

increased to 17%. Adsorption rate of Copper and Chromium time was 105min. It is also observed that the experimental results of adsorption isotherms fit well for Langumir model and maximum adsorption capacity is 3.15 mg/g and 1.63 mg/g for Cu and Cr at alkaline pH. Formation of Monolayer adsorbate on the outer surface of adsorbent. Kinetic study showed that pseudo first order reaction fitted well, the results show that heavy metals are physisorbed. Hence, IONCS is a low cost and effective adsorbent for toxic heavy metals present in water and wastewater.

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