



A Comparative Study of the Adsorption Efficiency of the Newly Synthetic Nano Iron Oxide and Commercial Activated Charcoal Towards the Removal of the Nickel(II) Ions

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Abstract: The synthetic nano iron oxide (SNIO) was synthesized by acid base hydrolysis and characterized by the XRD, SEM and EDAX techniques. Batch adsorption experiments were carried out to study the sorption behaviour of SNIO and commercial activated charcoal (CAC) towards Ni(II) ions as a function of initial concentration of the adsorbate, adsorbent dosage, contact time and pH. The adsorption for Ni(II) is found to be better in acidic pH for both SNIO and CAC. The equilibrium adsorption isotherm data have been tested by applying both Freundlich and Langmuir isotherm models. The Separation factor R was found to be between 0 and 1 for both the adsorbent, it clearly indicates the feasibility of adsorption.

Keywords: Adsorption, Synthetic nano iron oxide (SNIO), Commercial activated charcoal (CAC), Ni(II) ion.

Introduction

Heavy metals such as cadmium, lead, nickel, mercury, cobalt and barium are commonly detected in material-manufactured industrial effluents. Out of these, nickel and its nickel compounds have many industrial and commercial applications. Ni(II) ion play a vital role for the function of all living organisms. However higher concentrations in both anthropogenic release and naturally varying levels may be toxic to living organisms¹⁻³. Inhalation of Ni(II) ion exposure in occupational settings is a primary route for nickel-induced toxicity, and it may cause toxic effects in the respiratory tract and immune system. Nickel dermatitis produces erythema, eczema and lichenification of the hands and other parts of the skin that contact with nickel ion. Nickel hypersensitivity also causes asthma, conjunctivitis and inflammatory reactions⁴. The exposure of the general population to nickel pollutant takes

place mainly through oral intake, primarily through water and food, as a contaminant in drinking water and as a constituent or contaminant of food^{2,5}.

With the increase in environmental pollution, efforts are made to develop low-cost materials to remove contaminants from aqueous solutions. Currently, a wide range of physico-chemical adsorption and biological treatment methods are used for the removal of hazardous contaminants⁶⁻⁹. Recent development of nano-technique has shed some light on this field and chemically modified nanomaterials were efficiently used for the removal of aqueous contaminants based on liquid phase adsorption¹⁰. For instance, magnetic nano Fe_3O_4 has attracted much attention not only in the field of adsorption but also in the areas of medical care and magnetic sensing in the recent decades¹¹⁻¹⁴. It is believed that magnetic nano particles exhibit the finite-size effect or high ratio of surface-to-volume, resulting in a higher adsorption capacity for metal removal. In addition, the easy separation of metal loaded magnetic adsorbent from solution can be achieved using an external magnetic field. The results from various other research groups¹⁵⁻²⁰ have shown that magnetic nano structured particles Fe_3O_4 can be successfully applied to adsorb metal ions in which the combined technique of adsorption and magnetic separation holds the advantages of flexibility, eco-friendly characteristics and economic viability.

The present study mainly focuses the efficiency of the synthetic nano iron oxide (SNIO) in the removal of the metal ions compared to the extensively used commercial activated charcoal (CAC) with respect to the parameters such as adsorbent dosage, equilibrium time, pH of the solution, initial concentration of Ni(II) ion and the rate of kinetics of adsorption.

Experimental

Preparation of Synthetic nano Fe_3O_4

6.5 g of Ferrous Chloride and 4.8 g of Ferric Chloride were dissolved in 200 mL of distilled water and 10% Polyethylene glycol together and dispersed by ultra sonic stirring for 10 minutes. Then the mixture was heated to 75 °C. The pH value of the mixture was adjusted to about 11.5 by the drop wise addition of sodium hydroxide. The mixing was continued for 2 hours at 60 °C and then agitated at 80 °C for 30 minutes, purified and washed several times with distilled water at 80 °C¹⁵⁻²⁰.

Activation of the commercial activated charcoal

About 500 g of the Commercial Charcoal was digested in 100 mL of 4N Nitric acid and 300 mL de-ionized water for two hours at 90 °C and kept aside overnight. It was washed with boiling de-ionized water several times to remove the acid (tested with pH paper) and the metal ions present in the CAC. The washings were tested with Eriochrome Black T indicator which imparts wine red or blue colour to the solution depending on the presence or absence of the metal ions respectively. The CAC was dried in an air oven for about five hours. It was then stored in an air-tight wide mouth reagent container and used for adsorption studies.

Adsorbate

The synthetic effluent samples were prepared by dissolving the required amounts of nickel(II) sulphate in de-ionized water. Different concentrations of Ni(II) ions were then prepared by the progressive dilution of the stock solution.

Adsorption studies

Batch adsorption studies were performed by adding appropriate volumes of Ni(II) ion solution and de-ionized water to make the total volume of the solution to 100 mL for SNIO and 75 mL for CAC in a two separate 250 mL leak-proof reagent bottles. To this a definite amount of SNIO and CAC was added. The solutions were equilibrated to a pre-determined

period at 28 ± 1 °C in a mechanical shaker operating a speed of 135 ± 5 rpm. After the equilibration, the SNIO was conveniently separated via an external magnetic field and the solution was collected for the determination of Ni(II) ion concentration measurements using Atomic Absorption Spectroscopy (AA-6300, Shimadzu) in accordance with the standard procedure. In the case of CAC study, after the equilibration, the solution was filtered through Whatman No.40 filter paper. First 10 mL fraction of the filtrate was discarded. The rest of the filtrate was collected and estimated for Ni(II) ion using AAS. The percentage removal of Nickel at any instant of time was calculated using the equation

$$\text{Removal of Nickel (\%)} = \frac{C_o - C_e}{C_o} \times 100,$$

where C_o and C_e are the concentrations of Ni(II) at the start of the adsorption and at any instant of time respectively.

Results and Discussion

X-ray analysis

The obtained material was compared with JCPDS file No.39-1346. The mean crystallite size was determined using the Scherrer formula: $t = 0.9\lambda / (B \cos\theta)$, wherein t is the crystallite size, λ is the radiation wavelength, θ is the Bragg angle and B is the width of the corresponding peak at half maximum. The XRD pattern shows a formation of nanostructured Fe_3O_4 with FCC crystal structure. The crystallite size was estimated to be about 20 nm.

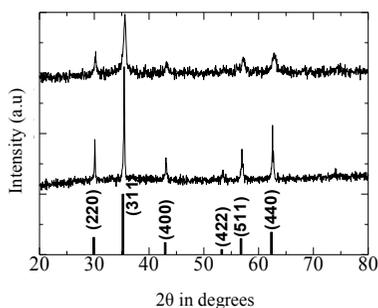


Figure 1. XRD pattern for synthetic nano Fe_3O_4 (SNIO), Commercial Fe_3O_4 and JCPDS file No. 39-1346.

SEM analysis

High resolution SEM images were made to examine the morphology and particle size of Fe_3O_4 , Ni(II) ion treated with Fe_3O_4 and CAC are given in the (Figure 2a-2d). The image of SNIO shows well defined particles in the range of 20-25 nm with mild agglomeration. It is comparable with the particle size obtained from the Debye-Scherer formula. However, after the adsorption of the nickel ion, the surface of the particles is modified and appears to be smooth with some agglomeration. The CAC shows bigger particles in the range 50–60 micron size with porous in nature. An x-ray energy dispersion analysis (EDAX) of the composition of the Fe_3O_4 and Ni adsorbed are shown in the (Figure 3a and 3b). The pattern shows the presence of iron and oxygen in the nano Fe_3O_4 and the nickel is identified along with iron and oxygen when nickel solution is treated with Fe_3O_4 . The BET surface area was measured for the SNIO and CAC using nitrogen as an adsorbate. The SNIO shows a surface area of $63.86 \text{ m}^2/\text{g}$ and the CAC shows about $1.37 \text{ m}^2/\text{g}$. This high surface area of SNIO is very useful for an effective adsorption of heavy metal ions.

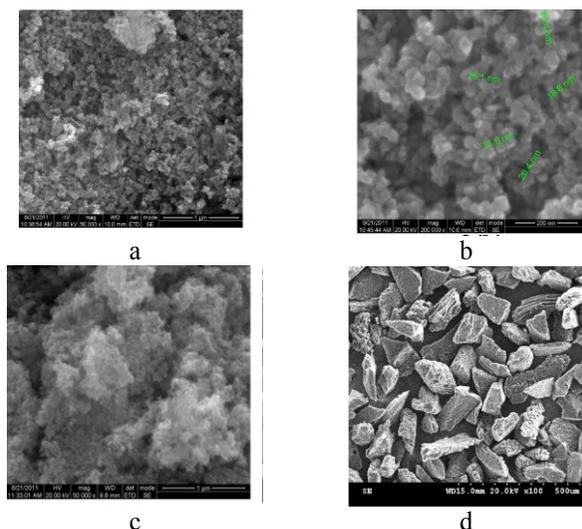


Figure 2. SEM micrograph for 2a) Synthetic nano Fe_3O_4 (SNIO) 2b) Magnified form of Synthetic nano Fe_3O_4 (SNIO) 2c) Nickel adsorbed in synthetic nano Fe_3O_4 2d) CAC.

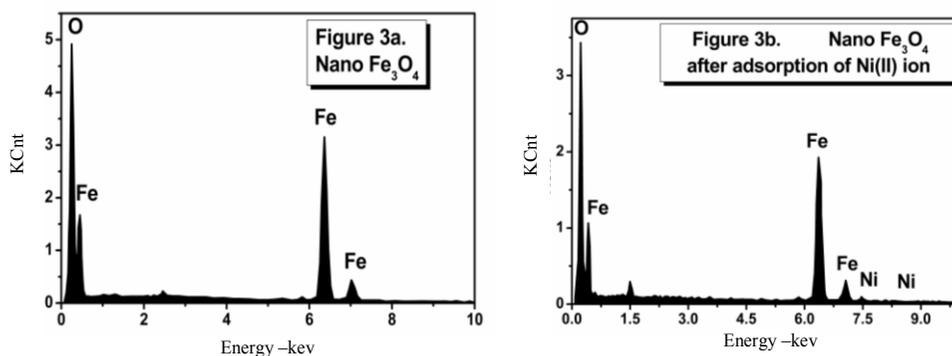


Figure 3. EDAX for a) Synthetic nano Fe_3O_4 (SNIO) b) EDAX for Synthetic nano Fe_3O_4 (SNIO) after adsorption of Ni(II) ion.

Effect of the initial concentration of the adsorbate

Initial concentration is one of the important factors for determining the adsorption capacity of an adsorbent. The adsorption experiment was carried out varying the concentration of Ni(II) ion (25, 50, 150, 200, 250, 300, 350 and 400 ppm) keeping the fixed dose of SNIO 2.57 g/L and CAC 13.3 g/L, contact time 60 minutes, pH 5-6.5 and temperature 28 ± 1 °C. The variations in the percentage removal of Ni(II) ion with its concentration are shown diagrammatically in (Figure 4). In both cases of adsorbents it was observed that the percentage removal of Ni(II) ion is low at higher concentration and gradually increases as the concentration of Ni(II) ion decreases. This is due to the fact that after the formation of mono-ionic layer at lower concentration over the adsorbent surface, further formation of the layer is highly hindered at higher concentration due to the interaction between Ni(II) ions present on the surface and in the solution. In addition to that, at low concentration of the Ni(II) ion, the ratio of the initial number of moles of the Ni(II) ions to the available surface area of the adsorbent is large and subsequently, the fraction of the adsorption becomes independent of the initial

concentration of the metal ion. But at higher concentration, the adsorption sites available for the adsorption become lesser and hence, the percentage removal of the metal ions at higher concentration decreases^{21,22}. The optimum concentration of the Ni(II) ion is found to be 50ppm for its efficient removal by adsorption on the SNIO. For CAC the optimum concentration of the Ni(II) ion is found to be 29.7 ppm. The percentage removal of the Ni(II) ion by SNIO at 50 ppm level is found to be 98.8%, whereas the CAC removes only 80.2% of the Ni(II) ion at 29.7 ppm concentration of the Ni(II) ion. The higher adsorptive power of the SNIO over the CAC may be attributed to the nanosize of the SNIO. When a particle shrinks to the nanometer range, an increasing fraction of the atoms are exposed to the surface, giving rise to excess surface energy compared to macro molecules. Therefore, nanoparticles with a higher total energy should be prone to adsorb molecules onto their surfaces in order to decrease the total free energy²³.

Effect of the dose of the adsorbent

The removal of the Ni(II) ion was studied with different dose of SNIO 0.82 – 4.57 g/L and CAC 1.33 -18.6 g/L at the optimum concentration of 50 ppm (SNIO) and 29.7 ppm (CAC) with fixed contact time 60 minutes and pH 5-6.5 and temperature 28±1 °C. The effect of the dose rate of SNIO and CAC on the removal of Ni(II) ion is pictured in (Figure 5). It is noted that the percentage removal of the Ni(II) ion increases as the adsorbent concentration increases owing to the enhanced total surface area of the adsorbent. The removal of the Ni(II) ion increases slightly with increase in the dose of the SNIO and CAC. This means that the toxic ions can be removed effectively from the contaminated water with the proper amount of the adsorbent, which would possess more adsorption sites available for the metal ion uptake from the solution^{21,22}. The adsorption capacity of SNIO is 98.8% but CAC shows only 80.2%. The SNIO shows higher adsorption capacity (98.8%) with the optimum dose of 2.57 g/L which is nearly five times less than the CAC (80.2% at 13.3g/L), because the nanoparticles possess more number of micropores and mesopores and higher total surface energy than macroparticles which are more prone to adsorb the Ni(II) ion on to the surface of the adsorbent in order to decrease the total energy²³.

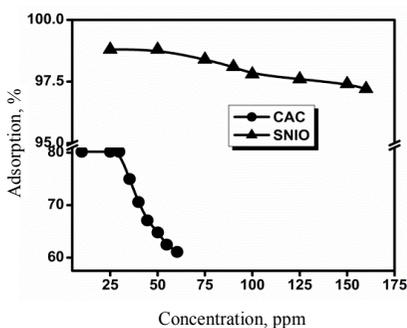


Figure 4. Variation in the percentage removal of the Ni(II) ion by CAC and SNIO with change in the initial concentration (dose rate: 13.3 g/L CAC, 2.57 g/L SNIO; contact time: 60 min; pH 6-6.5).

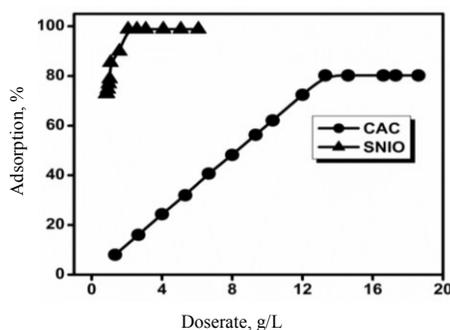


Figure 5. Variation in the percentage removal of the Ni(II) ion by CAC and SNIO with the dose rate of the CAC and SNIO (Ni(II) ion optimum concentration: 29.7 ppm (CAC), 50ppm(SNIO); contact time: 60min; pH 6-6.5).

Effect of the contact time

Contact time is another effective factor in batch adsorption technique. In order to study the effect of contact time on the removal of the Ni(II) ion, experiments were conducted at different contact times from 2 to 80 minutes keeping the optimum concentration of 50 ppm for SNIO and 29.7 ppm for CAC, optimum dose of SNIO 2.57 g/L and CAC 13.3 g/L, pH 5-6.5 and temperature 28± °C. The variation of the percentage removal of the Ni(II) ion by the

adsorption of CAC and SNIO with contact time is shown in (Figure 6). The extent of removal of the Ni(II) ion increases initially and then stagnant after the optimum contact time. For SNIO the optimum contact time was found to be 10 minutes for the effective removal (98.8%) of Ni(II) ion, whereas for the CAC the optimum contact time was 60 minutes for the removal of 80.2% of the Ni(II) ion. Within 10 minutes the SNIO shows higher adsorptive power than the CAC because the former possesses large surface area ($63.87 \text{ m}^2/\text{g}$), whereas the latter possesses small surface area ($1.37 \text{ m}^2/\text{g}$).

Effect of the pH

The pH is another important parameter controlling the uptake of Ni(II) ion from the aqueous solution by the adsorbent. The adsorption potential of the SNIO and CAC was found out at various pH values (1.8 to 8) keeping the system at the following conditions: initial concentration of the Ni(II) ion 50 ppm for SNIO and 29.7 ppm for CAC, optimum dose of 2.57 g/L for SNIO and 13.3 g/L for CAC, contact time 10 minutes for SNIO, and 60 minutes for CAC. The plots of the percentage removal of Ni(II) ion with respect to pH change was shown in (Figure 7).

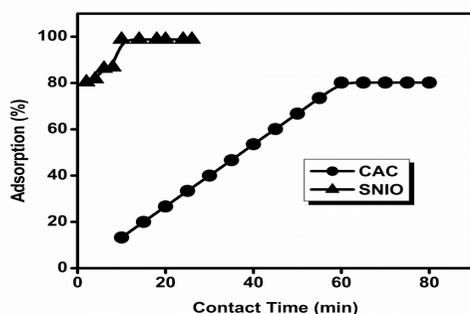


Figure 6. Variation in the percentage removal of the Ni(II) ion by CAC and SNIO with change in contact time (Ni(II) ion optimum concentration: 29.7 ppm (CAC), 50ppm(SNIO) ; optimum dose rate: 13.3 g/L CAC, 2.574g/L SNIO; pH 6-6.5).

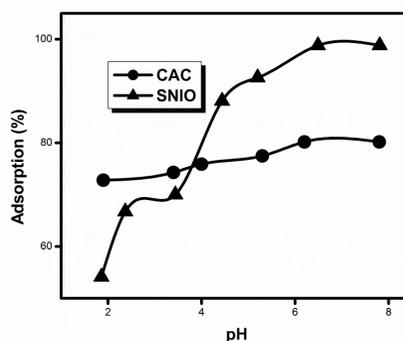


Figure 7. Variation in the percentage removal of the Ni(II) ion by CAC and SNIO with change in pH (Ni(II) ion optimum concentration: 29.7 ppm (CAC), 50ppm (SNIO) ; optimum dose rate: 13.3 g/L CAC , 2.574g/L SNIO ; optimum contact time:60min (CAC), 10 min (SNIO).

The removal of the Ni(II) ion through adsorption by the SNIO and CAC is effective in slightly acidic medium and the optimum pH range observed for both the SNIO and CAC is same (6-6.5). The percentage removal of metal ions is low at low pH and increases with the increase in pH. This observation reveals that the adsorption process is pH dependent. At low pH, because of the higher concentration of the H^+ ion and its higher mobility due to smaller size, the H^+ ions are preferentially adsorbed. Below pH_{ze} (point of zero charge) the surface of the adsorbent is positively charged and prevents the adsorption of the positively charged Ni(II) ion. At optimum pH (6-6.5), the concentration of the H^+ ions is lowered and consequently, the adsorption of the Ni(II) ions increases. At higher pH than the optimum pH, the OH^- ion concentration is increased and preferentially adsorbed on the adsorbent and the surface of the adsorbent becomes negatively charged. Moreover at higher pH, the Ni(II) ion form various complex anion, hydroxide

complexes, *etc.* which are retarded by the negatively charged surface of the adsorbent²⁴. At optimum pH the SNIO shows higher adsorption capacity of 98.8% whereas the CAC shows only 80.2%.

Kinetics of the adsorption

In order to find out the nature and order of kinetic of adsorption in the present study the applicability of the Natarajan & Khalaf²⁵, Legergren equations²⁶ is tested. The above equations are based on the fact that the adsorption follows first order kinetics. A linear relationship is found out with each equation as shown in (Figure 8). Hence, it is concluded that in the present study, the adsorption follows the first order kinetics. The rate constants computed using the above equations are given in (Table 1). The rate constant for the adsorption of Ni(II) ion on SNIO is found to be greater than CAC. The reason for this may be due to the large surface area and the highly active surface sites present in the SNIO than in CAC. Apart from the adsorption at the outer surface of the adsorbent, there also exists a possibility of inter-particle diffusion of the adsorbate molecule from the bulk of the outer surface into the internal pores of the adsorbent. The possibility was explored by plotting the amount of Ni(II) ion adsorbed (x/m) per unit mass of the adsorbent against $time^{1/2}$. The linear plots was observed and shown in (Figure 9), indicate that the intra-particle diffusion is the rate limiting step. The greater the intra-particle diffusion co-efficient, greater is the rate of adsorption. The rate of adsorption is higher in the case of SNIO than in the CAC.

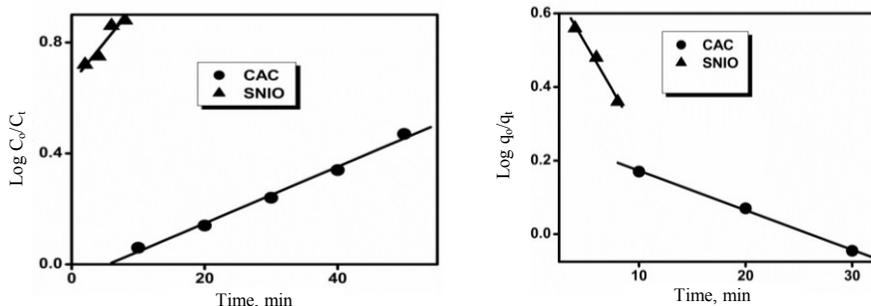


Figure 8. Natarajan and Khalaf and Legergren equation for the removal of Ni(II) ion by CAC and SNIO at different time interval.

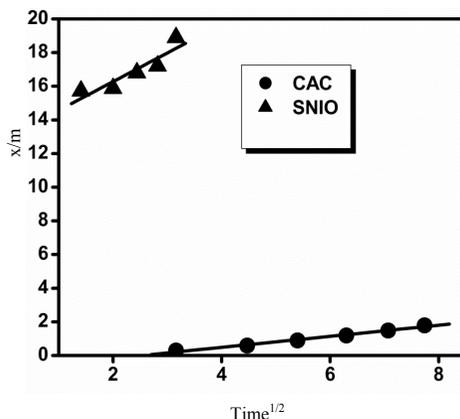


Figure 9. Intra Particle equation for the removal of Ni(II) ion by CAC and SNIO at different time interval.

Table 1. Rate constant of the adsorption of the Ni(II) ion by CAC and SNIO at different time interval.

Adsorbent	Rate constant of adsorption on CAC and SNIO, 10^{-2} min^{-1}			Intra particle diffusion coefficient $K_p, 10^{-2} \text{ min}^{-1/2}$
	K_a^1	K_b^2	Average	
CAC	1.0	3.0	2.0	32
SNIO	2.3	11	6.65	170

¹Natarajan and Khalaf equation, ²Legergren equation.

Modelling of the adsorption

The most common empirical models used for the adsorption equilibrium of the metal ions are due to Freundlich and Langmuir^{27,28}. The data obtained in the adsorption studies are analyzed by the classical Freundlich and Langmuir equations (Figures 10). In order to find out the most appropriate model for the adsorption of the Ni(II) ions, the data are fitted to each isotherm model. The essential parameters like adsorption capacities of the adsorbent Q_0 and k , the energy of the adsorption b , the separation factor R and the adsorption intensity $1/n$ obtained from the above models are given in (Table 2). The results show that the Freundlich and Langmuir adsorption isotherms are the best model for the adsorption of Ni(II) ion on the SNIO and CAC with the correlation coefficient (r^2) of 0.94 and 0.99. Adsorption capacity is found to be high for SNIO ($Q_0 = 111.1$) than CAC ($Q_0 = 3.57$), and the energy of the adsorption is found to be less for SNIO ($b = 0.04$) than CAC ($b = 0.13$). The low energy of the adsorption for the SNIO may be due to the larger surface area and the highly active surface sites present in the SNIO than CAC. The separation factor R obtained for the removal of Ni(II) ion by the SNIO (0.3) and the CAC (0.2) are found to be between 0 and 1 indicating the feasibility of the adsorption. The adsorption intensity $1/n$ values for the SNIO and the CAC are > 0.05 indicating a strong bond formation between the adsorbate and the adsorbent during the adsorption²⁹.

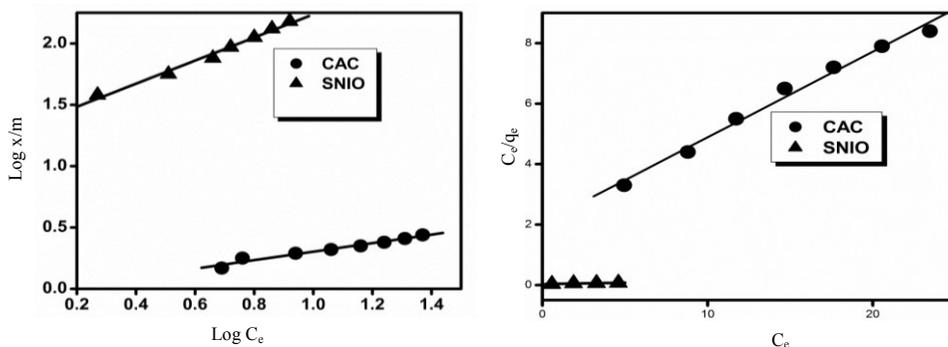


Figure 10. Application of Freundlich and Langmuir isotherm for removal of the Ni(II) ion by CAC and SNIO.

Table 2. Freundlich and Langmuir constants derived from the simultaneous removal of Ni(II) ion by using CAC and SNIO.

Adsorbent	Freundlich isotherm			Langmuir isotherm			
	K	1/n	r^2	Q_0, mgg^{-1}	B, Lmg^{-1}	R	r^2
CAC	0.91	0.3	0.99	3.57	0.13	0.2	0.99
MNIO	19.49	0.9	0.99	111.1	0.04	0.3	0.94

Conclusion

In the present study, the efficiency of the SNIO towards the removal of the Ni(II) ion was examined and compared with that of the CAC. The optimum conditions of the various factors for the maximum removal of the Ni(II) ion arrived at from the studies is given in (Table 3). The kinetic study showed that the removal of the Ni(II) ion on the SNIO and CAC followed first-order kinetics and the rate constant were found to be $2 \times 10^{-2} \text{ min}^{-1}$ and $6.65 \times 10^{-2} \text{ min}^{-1}$ respectively. The energy of the adsorption for the SNIO was found to be lower compared to the CAC. The higher adsorption capacity, higher rate constant of adsorption and lower energy of the adsorption of the SNIO compared to the CAC attributed to the large surface area and higher active surface sites of the SNIO. Hence, the SNIO may be considered to be an alternative adsorbent for the removal of the Ni(II) ion than CAC.

Table 3. The optimum conditions of the various factors for the maximum removal of the Ni(II) ion arrived at from the studies are.

Parameter	SNIO	CAC
Initial concentration of the Ni(II)ion, ppm	50	29.7
Dose rate, g/L	2.57	13.3
Contact time, min	10	60
pH	6-6.5	6-6.5
Percentage Removal, %	98.8	80.2
Adsorption capacity, mg/g	111.1	3.57
Energy of adsorption, L/mg	0.04	0.13

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