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# REMOVAL OF HEAVY METAL Ni (II) AND Cr (VI) FROM AQUEOUS SOLUTION BY SCOLECITE NATURAL ZEOLITE

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## ABSTRACT

The batch removal of Ni (II) and Cr (VI) ion from aqueous model solution using scolecite natural zeolite (SNZ) has been investigated under different operational parameters like initial heavy metal ion concentration, adsorbent amount, effect of pH and reuse of adsorbent amount. Natural zeolite used is characterized by SEM with EDX analysis. The equilibrium adsorption capacity of SNZ adsorbent used for Ni (II) and Cr (VI) were measured and the experimental data was analyzed by using Langmuir and Freundlich isotherm models and the experimental data are found to fit with Freundlich isotherm. The adsorption selectivity of zeolite for chosen heavy metal is Cr > Ni.

**Key words**: Scolecite natural zeolite, Adsorption, Langmuir isotherm, Freundlich isotherm.

## INTRODUCTION

Heavy metals are elements having atomic weights between 63.5 and 200.6, and a specific gravity greater than 5.0[1]. With the rapid development of industries such as metal plating facilities, mining operations, fertilizer industries, tanneries, batteries, paper industries and pesticides, etc., heavy metals wastewaters are directly or indirectly discharged into the environment increasingly, especially in developing countries. Unlike organic contaminants, heavy metals are not biodegradable and tend to accumulate in living organisms and many heavy metal ions are known to be toxic or carcinogenic. Toxic heavy metals of particular concern in treatment of industrial wastewaters include zinc, copper, nickel, mercury, cadmium, lead and chromium.

Now-a-days heavy metals are the environmental priority pollutants and are becoming one of the most serious environmental problems. So these toxic heavy metals should be removed from the wastewater to protect the people and the environment. Many

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methods that are being used to remove heavy metal ions include chemical precipitation[2], sulfide precipitation[3] investigated pyrite and synthetic iron sulfide for removal of lead and copper. Ion-exchange processes have been widely used to remove heavy metals from wastewater due to their many advantages, such as high, treatment capacity, high removal efficiency and fast kinetics[4-6]. Adsorption additives[7], tannic acid[8], magnesium[9], surfactants[10] and AC composite could be effective adsorbents for heavy metals. Agricultural waste material as potential adsorbent for sequestering heavy metal ions from aqueous solutions[11], membrane filtration[12] NF used for nickel[13,14] performed a new working system to investigate the removal of hexavalent chromium ions using electrodialysis electrochemical treatment technologies, etc.[15] studied the performance of an EC (electrochemical treatment technologies) system with aluminium electrodes for removing of metal ion from water.

Various types of adsorbent used in heavy metal removal are activated carbon[16], clay minerals[17,18], biomaterials[19], zeolites[20,21] and some industrial solid wastes[22,23] have been widely used as adsorbents for adsorption of ions and organics in wastewater treatment. Since the original discovery in this paper adsorption study has done for removal of Ni and Cr by using natural zeolite as an adsorbent. As adsorption is now recognized as an effective and economic method for heavy metal wastewater treatment. The adsorption process offers flexibility in design and operation and in many cases will produce high-quality treated effluent. In addition, because adsorption is sometimes reversible, adsorbents can be regenerated by suitable desorption process.

Zeolite is an aluminum silicate that occurs both as natural and as produced synthetic. Zeolite has a three-dimensional structure with pores. It consists of silicon, aluminum and oxygen ions. The silicon ions are neutrally charged in the crystal structure. Aluminum ions create negative places. To keep the cargo in balance a counter ion ($Na^+$, $K^+$) or a proton ($H^+$) is present in the pores. One types of zeolite have just as large pores through the entire crystal structure. The crystal structure is stipulated by the ring size of the crystal structure. To change the ratio of aluminum on silicon can also influence the pore size as well as the type of a counter ion. All natural zeolites contain aluminum and are hydrophilic in nature[24].

Natural zeolites, which are aluminosilicate minerals and found in volcanogenic sedimentary rocks, possess several important properties including adsorption, cation-exchange, dehydration–rehydration, and catalysis. These natural zeolites have broad range of applications in construction materials, soil improvements for water and nutrient retention, treatment of water and wastewater for the removal of heavy metals and nutrients.

Natural zeolite used in this study is considered as a low-cost adsorbent material, which is easily available in much in western region of Maharashtra (India). The samples were collected from naturally occurring zeolite around Poona region, India. Scolecite is a calcium zeolite having the chemical formula $CaAl_2Si_3O_{10}\cdot 3H_2O$ (hydrous calcium aluminosilicate). It has a non-metallic, glassy luster, is variably colored, has a white streak, is moderately hard (H = 5 to 5.5), and often forms radiating clusters of acicular crystals**.**

Our objective in this study is to investigate the removal of inorganic pollutants like Ni (II) and Cr (VI) ions from a model solution by the Scolecite natural zeolite (SNZ). Studied the influence of operational parameters such as metal ion concentration and amount of adsorbent. The Langmuir and Freundlich models are used to fit the model isotherm. In this study these two metals are selected because nickel exceeding its critical level might bring about serious lung and kidney problems aside from gastrointestinal distress, pulmonary fibrosis and skin dermatitis[25]. It is known that nickel is human carcinogen also chromium exits in the aquatic environment mainly in two states: Cr (III) and Cr (VI). In general, Cr (VI) is more toxic than Cr (III). Cr (VI) affects human physiology, accumulates in the food chain and causes severe health problems ranging from simple skin irritation to lung carcinoma[26].

## Materials and methods

### Materials

The natural zeolite sample of scolecite was collected from west part of Maharashtra (India). It is crushed up and sieved to the particle size in of 32-45 nm. This powder sample used for experimental study as it is without any wash. Stock solution of Ni (II) and Cr (VI) containing 1000 mg of metal ion were prepared by dissolving appropriate amounts of nickel nitrate ($Ni(NO_3)_2.6H_2O$) and potassium chromate ($K_2CrO_4$) compound separately in distilled water and this stock solution was diluted appropriately as necessary.

### Characterization

The crystal structure and morphology of zeolite particles obtained by characterization using scanning electron microscopy (SEM, JEOL JSM-5600LV, 20 KV).

### Batch adsorption studies

### Effect of initial heavy metal ion concentration

Adsorption studies onto the natural zeolite material were conducted in glass beaker using a constant mass of the adsorbent 1 g/100 mL (10 g/L) for different ion concentration like (10, 20, 30, 40, 50, 60 mg/L)). Mixer stirred for 2 hr at room temperature by magnetic stirrer at constant speed and samples are collected after some time interval. Then suspensions were filtered by using centrifuge and this filtrate sample were analyzed for ion concentrations by Atomic Absorption spectroscopy (AAS).

## Effect of adsorbent amount

Different mass quantities of the adsorbent like (1, 1.5, 2, 2.5, 3 g/100 mL ion solution) were mixed with 0.1 L of 20 mg $L^{-1}$ (20 ppm) solution of potassium chromate and nickel nitrate aqueous solutions. Then heavy metal ion salt and adsorbent are stirred by magnetic stirrer for 2 h at room temperature and samples are collected after some time interval. Then suspensions were filtered by using centrifuge and this filtrate sample were analyzed for ion concentrations by AAS.

## Effect of pH

Adsorption of heavy metal ion on SCZ has studied at various pH values like (3, 5, 7, 9, 11) with adsorbent amount of 10 g/L, metal ion concentration 20 mg/L, temperature 26°C, interaction time 120 min. with constant magnetic stirring.

## Effect of reuse of adsorbent amount

Adsorption of heavy metal ion on SCZ has studied by reuse of adsorbent amount several times. With adsorbent amount of 10 g/L, metal ion concentration 60 mg/L, temperature 26°C, interaction time 60 min. with constant magnetic stirring. In first run of experiment adsorbent and heavy metal ion solution are stirred with constant magnetic stirring for 1 hr after that sample are collected and filtered by centrifuge for AAS analysis and remaining sample in beaker are filtered through the Whatman filter paper and washed 3-4 time with distilled water. Collected adsorbent amount are further dried for 12 hour in electric oven at 60°C temperature. This dried adsorbent powder sample are further used for 2nd run of experiment with keeping all other parameters and operational process same. This same process is carried out for seven runs of experiment.

## Adsorption isotherm

The equilibrium distribution of metal ions between the sorbent and the solution is important in determining the maximum sorption capacity. Several isotherm models are available to describe the equilibrium sorption distribution in which two models are used to fit the experimental data: Langmuir and Freundlich models. The linear form of Langmuir model[27] is given by -

$$C_e/q_e = (1/Q_{max}.K_L) + (C_e/Q_{max})$$

Where, $q_e$ is metal concentration on the zeolite at equilibrium (mg of metal ion/g of zeolite); $Q_{\max}$ (mg/g) and $K_L$ (1/mg) are Langmuir constants related to the maximum adsorption capacity corresponding to complete coverage of available adsorption sites and a measure of adsorption energy (equilibrium adsorption constant), respectively. These constants are found from the slope and intercept of $c_e/q_e$ vs. $C_e$ linear plot so that $Q_{\max}$ = 1/ slope and $K_L$ = slope/intercept. The linear form of the Freundlich model[27] is also given by-

$$\text{Ln } q_e = \ln K_F + (1 /n) \ln C_e$$

Where, $K_F$ and $n$ are Freundlich constants determined from the slope and intercept of plotting ln $q_e$ vs. ln $c_e$. Amount of metal ion adsorbed on natural zeolite is calculated at the difference between initial and final concentrations at equilibrium.

$$q_e = (C_i\text{-}C_e)/S$$

Where, $q_e$ is the ion concentration adsorbed on the SNZ at equilibrium (mg of ion/g of SNZ), $C_i$ is the initial concentration of ion in the solution (mg/L) and $C_e$ is the equilibrium concentration or final concentration of ions in the solution (mg/L). The does (slurry) concentration, $S$, is expressed by,

$$S = m/V$$

Where, V is the initial volume of ions solution used (L) and $m$ is the mass of SNZ used (g). The percent adsorption (%) was calculated using the following equation.

$$\% \text{ adsorption} = ((C_i\text{-}C_e)/ C_i) *100\%$$

# RESULTS AND DISCUSSION

## Characterization of adsorbent

## SEM with EDX study

SEM photograph of zeolite particles is shown in Fig. 1. It is clear that particles are rectangular in shape with sharp edges. The qualitative composition has obtained by Energy-dispersive X-ray spectroscopy (EDX) analysis can be seen in Fig. 2. All composition has given in Table 1. EDX analysis of adsorbent gives the result of chemical compositions present in zeolite along with peaks of all compounds as shown in Fig. 2. The SNZ were composed mainly of $SiO_2$ and $Al_2O_3$ components whose Si/Al ratio becomes 1.88 that means there are two Si atoms for each single Al atom. While the MnO and $K_2O$ contents were very low. The SNZ contained large amounts of CaO as compared with the other component, thus appears that a high CaO content might enable the formation of hydroxy sodalite. It has higher contents of calcium compound so it's called calcium zeolite.

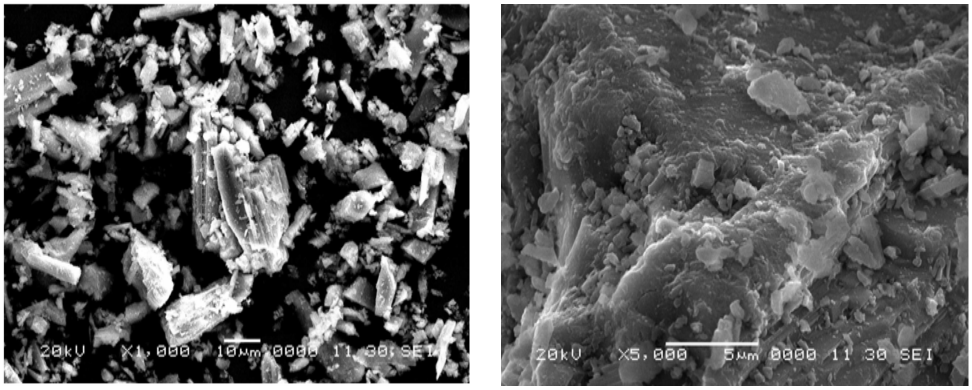


**Fig. 1: SEM photographs of SCZ (Scolecite natural zeolite)**

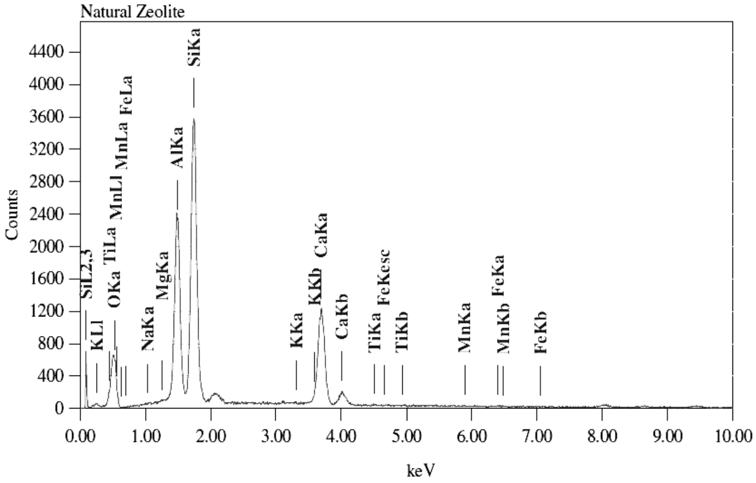


**Fig. 2: EDX of SCZ (Scolecite natural zeolite)**

**Table 1: Chemical compositions of natural zeolite obtained from EDX study**

| Compound | $Al_2O_3$ | $SiO_2$ | $K_2O$ | CaO | $TiO_2$ | MnO | FeO | $Na_2O$ | MgO |
|---|---|---|---|---|---|---|---|---|---|
| **Mass%** | 27.82 | 52.30 | 0.07 | 18.60 | 0.31 | 0.15 | 0.31 | 0.21 | 0.23 |

## Adsorption of heavy metal ion

### Effect of the initial heavy metal ion concentration

The adsorption of Ni (II) and Cr (VI) onto SNZ as a function of their concentrations was studied at room temperature by varying the heavy metal ion concentration from 10 to 60 mg/L. The results are shown in Fig. 3 and Fig. 4. The percentage removal efficiency for Ni (II) and Cr (VI) decreased with increasing metal concentration in the aqueous solutions. The number of metal ions adsorbed per unit mass ($q_e$) of adsorbent increases gradually with increase in metal concentration, whereas the extent of adsorption (%) decreases with increasing metal ion loading (Fig. 3A and 4A) of Cr (VI) and Ni (II) resp., For example, by changing the initial concentration of Ni (II) from 10 to 60 mg/L, $q_e$ increases from 0.85 to 3 mg/g. Whereas the extent of adsorption (%) decreases from 77.39 to 51.31%. Similar result observed in Cr (VI) adsorption. At low initial metal ion concentration, the ratio of the number of metal ions to the number of available adsorption sites is small and consequently the adsorption is independent of the initial concentration, but as the concentration of metal ions increases, the situation changes and the competition for adsorption sites becomes fierce[29]. At high concentration of metal, a unit mass of the adsorbent is exposed to a larger number of metal ions and a progressively higher number of metal ions are taken up with the gradual filling up of the appropriate binding sites.

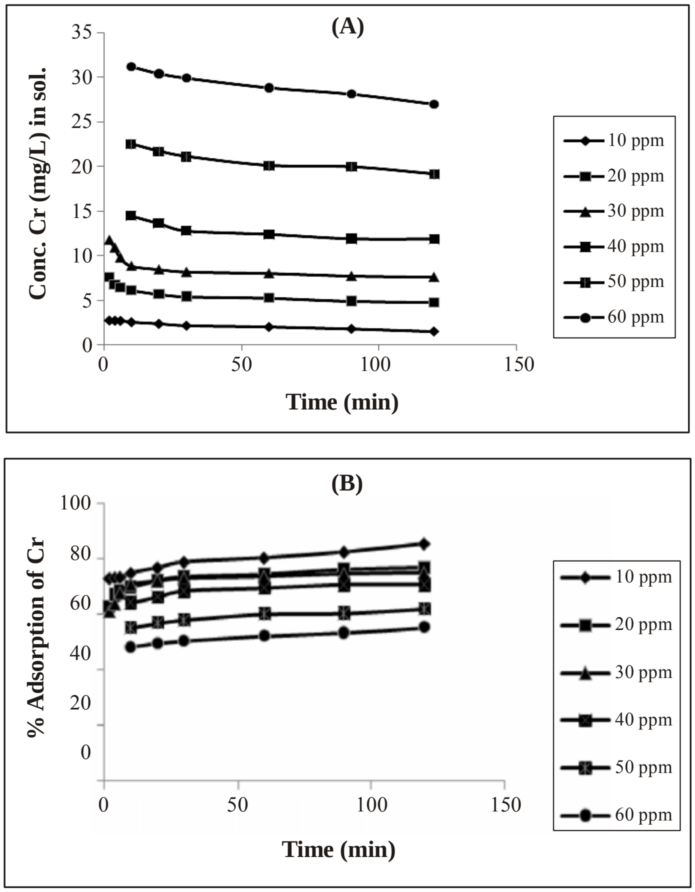


**Fig. 3: Effect of concentration on adsorption of Cr (VI) has shown in (A) and (B), resp.**

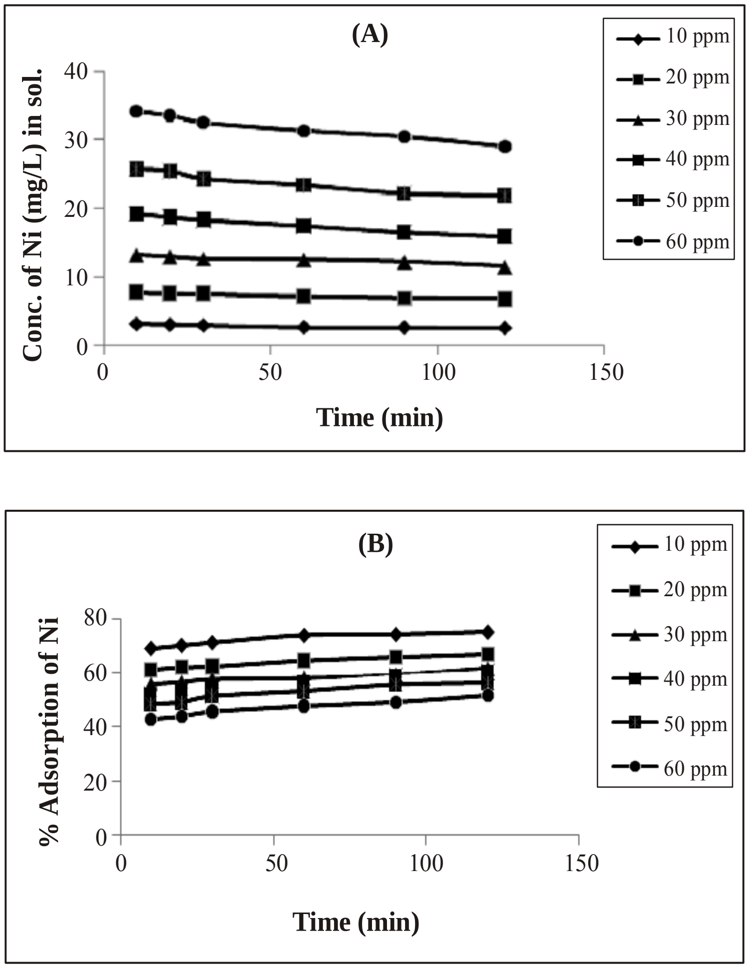


**Fig. 4: Effect of concentration on adsorption of Ni (II) has shown in (A) and (B), resp.**

## Effect of amount of adsorbent

The effect of the SNZ adsorbent dosage on the removal of Cr (VI) and Ni (II) has shown in Fig. 5(A) and Fig. 5(B), resp. Adsorbent dosage was varied from 10 to 30 g/L. The initial ion concentrations were 1000 (mg/L), stirring time was 2 hr and concentration of metal ion was 20 ppm (mg/L). In all the cases, the extent of adsorption (%) increases rapidly with increase in the adsorbent amount. This is to be expected because, for a fixed initial solute concentration increasing adsorbent amount provides greater surface area or adsorption sites[30]. However, the amount adsorbed per unit mass ($q_e$) decreases for selected metal ions. A large amount of adsorbent effectively reduces the unsaturation of the adsorption sites and correspondingly the number of such sites per unit mass comes down, resulting gives less adsorption at higher adsorbent amount. Ex. the amount of Cr (VI) adsorbed per unit mass of adsorbent ($q_e$) decreased from 1.53 to 0.601 mg/g and for Ni (II) it becomes 1.332 to 0.578 mg/g respectively, as the adsorbent amount was changed from 1.0 to 3.0 g/L. The corresponding extent of adsorption increased from 73.55 to 83.65 % for Cr (VI) and from 63.63 to 79.19 % in the same order for Ni (II) respectively. The variation in the extent of adsorption (%) and the amount adsorbed per unit mass ($q_e$) for adsorbent–metal systems has given in Table 2. Similar observations have been reported earlier for Ni (II) on montmorillonite[31].

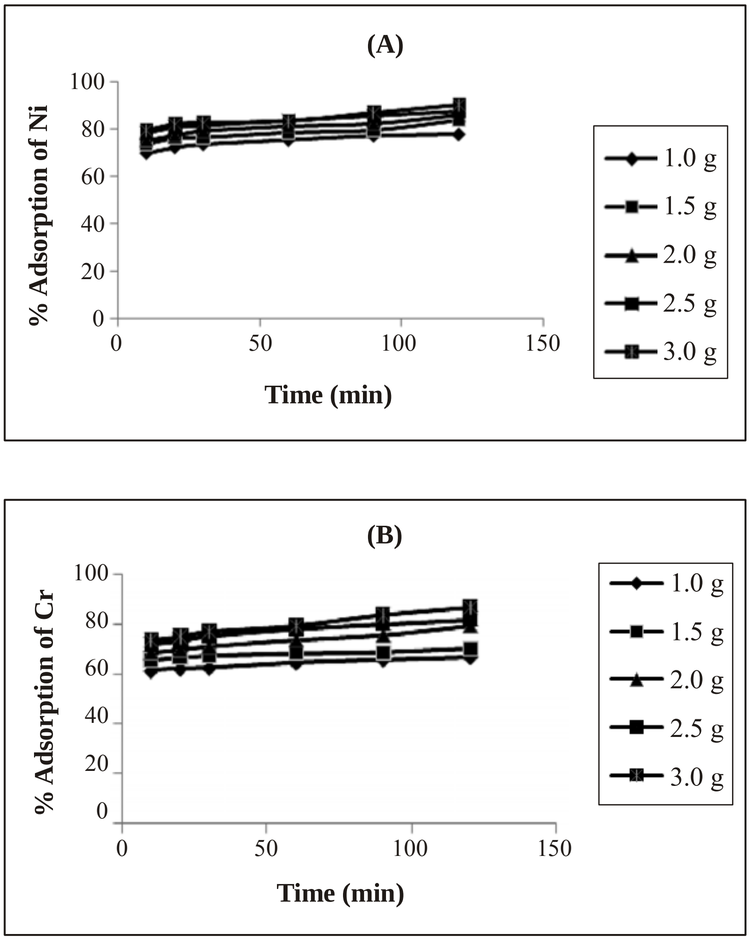


**Fig. 5: Effect of adsorbent (natural zeolite) amount on adsorption of Cr (VI) and Ni (II) has shown in (A) and (B), resp.**

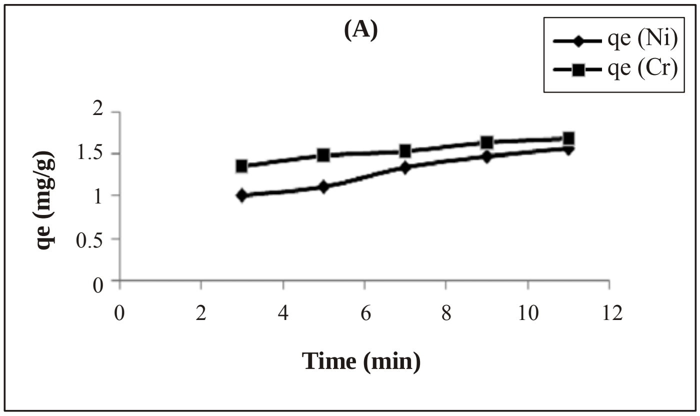
**Table 2: Effect of adsorbent amount on extent of adsorption (%) and amount of metal ion adsorbed per unit mass of adsorbent ($q_e$, mg/g) for adsorption of Cr (VI) and Ni (II) on natural zeolite (SNZ)**

| Adsorbent amount in (g/L) | Cr (VI) | | Ni (II) | |
|---|---|---|---|---|
| | % adsorption | $q_e$ (mg/g) | % adsorption | $q_e$ (mg/g) |
| **1.0** | 73.55 | 1.53 | 63.63 | 1.332 |
| **1.5** | 80.79 | 1.086 | 69.42 | 0.932 |
| **2.0** | 85.9 | 0.8485 | 76.9 | 0.793 |
| **2.5** | 87.88 | 0.6896 | 80.89 | 0.654 |
| **3.0** | 89.12 | 0.601 | 84.45 | 0.578 |

## Effect of pH

Adsorption of Ni (II) and Cr (VI) by SCZ could be studied over the pH range from 3-11 without any abrupt changes in adsorption. The general trend in the extent of adsorption (%) as well as amount adsorbed ($q_e$) with increase in pH is positive. Fig. 6(A) represents the trend in the amount of metal ions adsorbed ($q_e$) with respect to pH from Fig. 6(B). It is observed that adsorption increases with increase in pH value of heavy metal ion solution.

At low pH, the number of $H_3O^+$ ions exceeds several times than metal ions and the metal ions can hardly compete with $H_3O^+$ ions for the binding sites on the clay adsorbents. With an increase in pH, the concentration of $H_3O^+$ ions decreases and some of the sites become available for metal ions. As the acidity decreases, more and more $H_3O^+$ ions on the adsorbent clay surface are replaced by metal ions and metal species $Ni^{2+}$ and $NiOH^+$ [31].



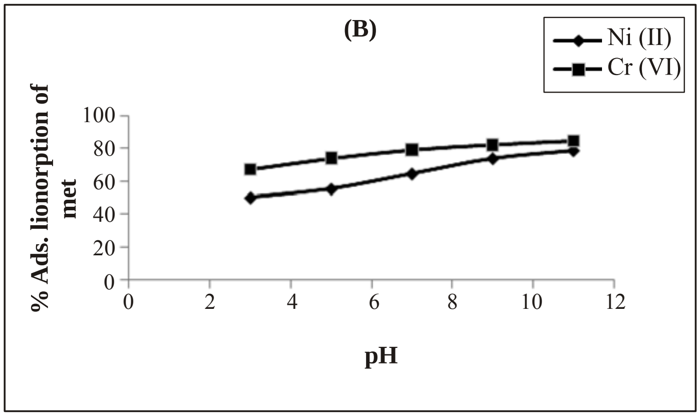


**Fig. 6: Influence of pH on the amount of metal ions adsorbed for adsorption of Cr (VI) and Ni (II) on SNZ (clay 10 g/L, initial metal concentration 20 mg/L, room temp.)**

## Reuse of Adsorbent amount

In the Study of reuse of adsorbent amount of 10 g/L for adsorption of 60 mg/L heavy metal ion concentration result is shown in Fig. 7, which represents that % adsorption decreases with increase in reuse of adsorbent. Adsorption of Cr (VI) decreases from 94.57 to 30.76 and that of Ni (II) decreases from 69.42 to 18.85 resp., zeolite can be reused four time to get 50% adsorption. Due to several washes with water it forms new sites for further adsorption, but due to same quantity of ion concentration for every run of experiments, adsorbents capacity of zeolite get reduced.

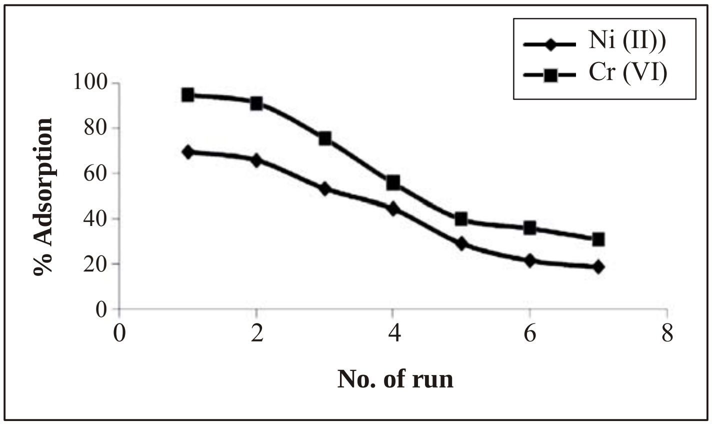


**Fig. 7: Effect of reuse of adsorbent amount on adsorption of Cr (VI) and Ni (II) on SNZ (clay 10 g/L, initial metal concentration 60 mg/L, 1 hr stirring at room temperature)**

## Adsorption isotherm

For initial metal ion concentrations of 10, 20, 30, 40, 50, 60 mg/L the plots of $q_e$ vs. $C_e$ for metal–adsorbent interactions for a fixed amount of adsorbent (10 g/L) at 303 K are presented in Fig. 8. It is seen that zeolite have lower uptake of Ni (II) than of Cr (VI). The empirical Freundlich isotherm yields linear plots ($r = 0.98$ for Cr (VI) and 0.99 for in Fig. 9) and the values of the coefficients, $n$ (all values $< 1.0$) and $K_F$ (0.7867 for Cr (VI) and 0.8112 for Ni (II)) indicate that the adsorbent have a good potential to be used as adsorbents for the metal ions Ni (II) and Cr (VI). It is to be noted that the Freundlich isotherm applies to adsorption on nonspecific and heterogeneous sites on solid surfaces, but no definite mechanism could be arrived at. The $K_F$ values of Ni (II) are higher than those of Cr (VI). This might be the case in the present work, since the Freundlich isotherm plots have good linearity.

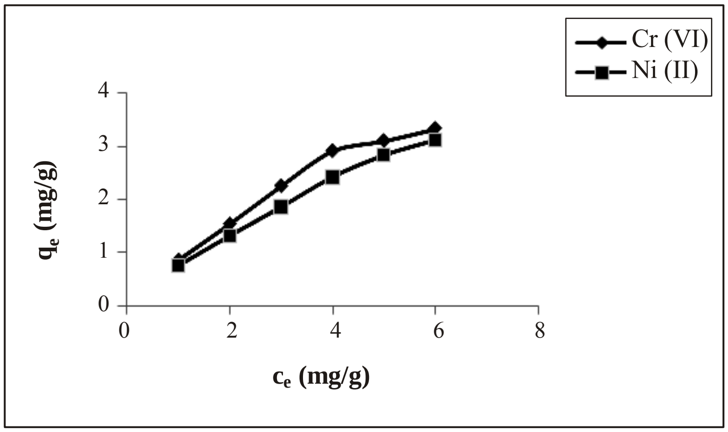


**Fig. 8: Isotherm plot of ($q_e$ vs. $c_e$) for adsorption of Cr (VI) and Ni (II) (adsorbent 10 gm/L, initial metal ion concentration 10, 20, 30, 40, 50, 60 mg/L, room temp., 2 hr constant speed stirring) at zeolite-metal equilibrium**

In Langmuir isotherm study Fig. 10 equilibrium coefficient, $K_L$, determines the direction in which the adsorbate–adsorbent equilibrium clay (solid phase) + metal (II) (aqueous phase) = clay–metal (II) is shifted. Large values of $K_L$ ensure that the equilibrium is predominantly driven toward the right, leading to the formation of the adsorbate–adsorbent complex. From the values of Langmuir plot. Got the equation of liner line for Ni (II) is [$c_e/q_e = 0.00094 + 0.1082\ C_e$], where value of equilibrium coefficient $Q_{max}$ is 9.242 and $K_L$ is 0.008568, resp. This suggests that the numbers of adsorption sites are less. Similarly for Cr (VI) equation of Langmuir linear plot is [$c_e/q_e = 0.00137 + 0.1185\ C_e$], where value of equilibrium coefficient $Q_{max}$ is 8.4388 and $K_L$ is 0.01158, resp. This suggests that the number of adsorption sites is less. So, Langmuir isotherm does not fit with the data.

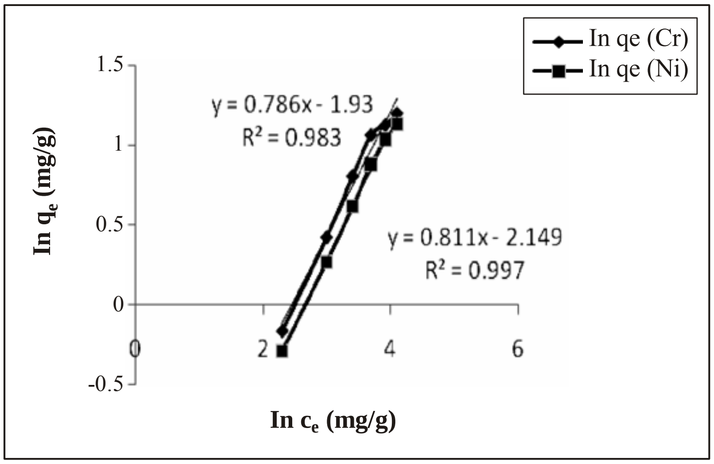


**Fig. 9: Freundlich Isotherm plot of ($\ln q_e$ vs. $\ln C_e$) for adsorption of Ni and Cr (VI) (adsorbent 10 gm/L, initial metal ion concentration 10, 20, 30, 40, 50, 60 mg/L, Room temp.) at zeolite –metal equilibrium**

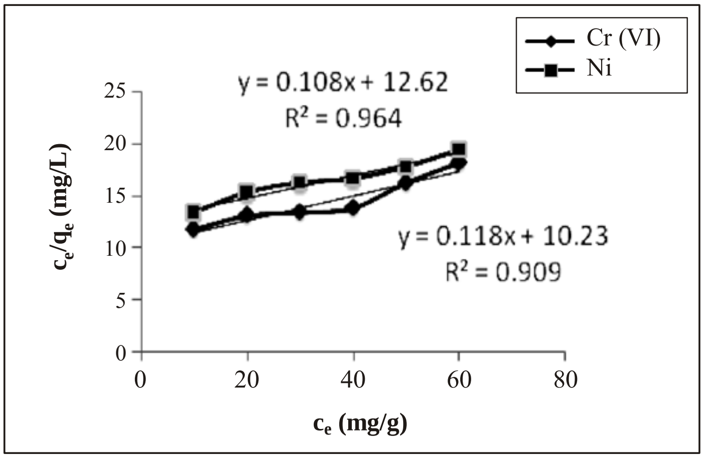


**Fig. 10: Langmuir Isotherm plot for adsorption of Ni and Cr (VI) (adsorbent 10 gm/L, initial metal ion concentration 10, 20, 30, 40, 50, 60 mg/L, room temp) at zeolite-metal equilibrium**

## CONCLUSION

In the present study, scolecite natural zeolites are capable of removing metal ions Ni (II) and Cr (VI) from aqueous solution. The maximum adsorption found at room temperature for metal ion concentration of 10 mg/L maximum adsorption removal of Ni (II) and Cr (VI) is 0.75 mg/g and 0.8515 mg/g, respectively by 10 g/L of zeolite quantity. The uptake of metal ions depends appreciably on the amount of adsorbent. The amount adsorbed per unit mass ($q_e$) and (%) adsorption show reverse trends with increase in adsorbent amount. pH effect show the ($q_e$) and (%) adsorption increase with increase in pH from (5-11) of salt ion solution. Zeolite can be reused successfully up to three runs with water wash. Both Langmuir and Freundlich isotherms yield good fits and Freundlich isotherm model provided best correlation of the equilibrium data ($R^2$ = 0.99) for Ni (II) and ($R^2$ = 0.98) for Cr (VI), respectively. Zeolite shows the selectivity as Cr (VI) > Ni (II).

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