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Synthesis of Iron Nanoparticles and Applications in the Removal of Heavy Metals from Aqueous Solutions

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Abstract

In this study, The synthesis of nano-size iron particles using a borohydride reduction of ferrous/ ferric ions in the presence ofsolvent containing ethanol, was investigated. The particle distribution of each sample of synthesized iron particles was characterized by powder X-raydiffraction XRD, which was used to determinethe prevailing phases.. Nanoscale iron particles have large surface areas and high surface reactivity. The size of the particles have been diagnosed by AFM, SEM, TEM microscopy.Then being used to remove elements (Cd ,Ni ,Cr) from water. At the adsorption process the element were determined by the percentage of adsorption in conjunction with atomic absorption spectroscopy ,also byEDX. The synthesized nano-ZVI material has shown aremarkable potential for heavy metals immobilization from aqueous water. Langmuir adsorption parameters for the adsorption of Cd, Cr, Ni were (k 0.85 , 0.71 , 0.67) .ΔH=11.6 , 22.8 , 16.6kJper mole.G=-12.6 , -15.5 , -12.9kJ per mole,ΔS=0.08 , 0.13 , 0.098kJ per mole respectively.

Keywords : Nanoparticles , Iron oxide , TEM , EDX , Heavy Metals, Adsorption

1.1: Introduction

Nanotechnology is the engineering and art of manipulating matter at the nanoscale (1–100 nm) [C. R. Keenan , D. L. Sedlak 2008]for environmental applications. Nanotechnology offers the potential of novel functional materials, processes and devices with unique activity toward complex contaminants, enhanced mobility in environmental media and desired application flexibility [Santra S, Tapec R2001]. Many nano-based environmental technologies (e.g., sensors, sorbents, reactants) are under very active research and development, and are expected to emerge as the next generation environmental technologies to improve or replace various conventional environmental technologies in the near future [N. C.Mueller, J. Braun and J. Bruns 2012]. The use of nanometals for subsurface remediation of chlorinated compound and heavy metal contaminated sites has received significant attention in part due to the ability of nanometals to rapidly transform contaminants in controlled laboratory experiments. Nanometals used for these purposes include nano iron and zinc, however nanoscale zero valent iron (nZVI) is most commonly used. In addition, other metals such as palladium or nickel have been added to increase the reduction rate. This combination of nZVI with a noble metal is referred to as a bimetallic nanometal [G.V. Lowry and K.M. Johnson.2004] The reactivity and the availability of existing technology to precisely design and synthesize nanometals make nanometals particularly attractive for the remediation of subsurface contaminants. This remediation technology involves a series of steps for nanometals . transport, in the aqueous phase (or other delivery fluid), to the contaminated zone . attachment to soils in the contaminated zone or partitioning to the NAPL/aqueous phase. reaction with the target contaminant to form less toxic or less mobile products laboratory experiments e.g. [Boparai 2011, Song 2005]. Nanometals have been proposed for use in the remediation of a variety of contaminants, including heavy metals. The transformation, solubility, mobility, and consequently toxicity, of heavy metals in the environment are governed by redox reactions, precipitation/ dissolution reactions, and adsorption/desorption phenomena.The solubility, mobility and toxicity of metals of environmental concern are strongly dependent upon their oxidation states. For example, at its higher oxidation state chromium (Cr^{6+}) is very toxic, whereas Cr^{3+}, an essential nutrient, is relatively non-reactive but can be toxic in large doses. Moreover, Cr^{6+} is highly soluble and mobile in soils whereas Cr^{3+} forms relatively insoluble oxide and hydroxide compounds [Jacobs JA,2005]. Given the strong dependence of Cr mobility and toxicity on its redox state, remediation technologies that reduce Cr^{6+}, such as a reduction by Fe0, are of significant interest.The specific removal mechanisms involved in treatment of heavy metal contamination with ZVI depend on the standard redox potential (E^0) of the metal contaminant , that of Fe0, (e.g., Cd and Zn) are removed purely by adsorption to the iron (hydr)oxide shell. Metals with E0 much more positive than Fe0 (e.g., Cr, As, Cu, U, and Se) are preferentially removed by reduction and precipitation [Li XQ, Zhang 2007]. Metals with slightly more positive E^0 than Fe^0, (e.g., Pb and Ni) can be removed by both reduction and adsorption. Oxidation and co-precipitation by iron oxides are the other possible reaction mechanisms depending upon the prevailing geochemical conditions such as pH, Eh and initial concentration and speciation of contaminant. if present in oxidized forms in solution, can be reduced by nZVI to create bimetallic nanoparticles (Fe^0/M^0) enhancing the rate of reaction of contaminants.[Babes L, Denizot B, Tanguy G and Le Jeune JJ,1999]

The adsorption process is primarily studied by fitting the data to Freundlich or Langmuir equations. The Freundlich isotherm is based on an empirical equation while the Langmuir equation is applicable only for a monolayer adsorption process [Khan, 2012]. Isotherm is the relationship between the equilibrium amount of adsorbate adsorbed on adsorbent surface and residual concentration of adsorbate in solution [Shirmardi et al., 2012].This isotherm describes adsorbate-adsorbent systems in which the extent of adsorbate coverage is limited to one molecular layer can be modeled by the Langmuir isotherm.The heat of adsorption is constant and independent of coverage (a consequence of no lateral interaction between adsorbate molecules).Each adsorbate molecule occupies only one site.The adsorption is localized (that is molecules remain at the site of adsorption until desorbed) [Barry and john, 1998].The expression of the Langmuir:

$$q = q_m \frac{K_L C_e}{(1 + K_e\ C_e)} \qquad (1\text{-}1)$$

q = The amount of adsorbate per gram of adsorbent (mg/g)
C_e= The equilibrium concentration of adsorbate in solution (mg/L)
K_L=The Langmuir constant (L/mg)
q_m= The theoretical saturation capacity of the monolayer (mg/g)
The values of (1/q_m= K_L=) and (1/qm) are calculated from the intercept and slope of the linear plot of $C_e/\,q$ versus C_e.[. Chingombe, B. Saha, R.J.2006]

$$\frac{\mathrm{C_e}}{\mathrm{q}} = \frac{1}{(K_L q_m)} + \frac{C_e}{q_m} \qquad (1\text{-}2)$$

The Freundlich model is an empirical equation that assumes heterogeneous adsorption due to the diversity of adsorption sites[G.Q. Tan, D. Xiao2009]. The Freundlich equation is
$q{=}K_F Ce^{1/n}$ (1-3)
q = The equilibrium of adsorbate concentration on adsorbent (mg/g).
Ce= The equilibrium concentration of adsorbate in solution (mg/L).
K_F,^{n} = The Freundlich constants, which represent the adsorption capacity and the adsorption strength [Chung, 2006].
This equation is conveniently used in the linear form by taking the logarithmic of both sides
$logq{=}logKF{+}1/nlogCe$ (1-4)
A plot of log (Ce) against log (q) yielding a straight line indicates the confirmation of the Freundlich isotherm for adsorption. The constants ($1/n$, KF) can be determined from the slope and the intercept [G.Q. Tan, D. Xiao 2009].
Mueller and his group prepared minutes iron zero nano iron Fe^0 and its applications to address soil and groundwater mainly from persistent chlorinated organic as well as inorganic ions or metal.(Mueller, N.C 2011)
Giasuddin and his group studied nzvi use in the removal, carb thioate pesticides, acids, humic and also TCE and other halogenated compounds from groundwater and soil water$^{(}$Giasuddin, A. B. M.; Kanel, S. R. and Choi, H.2007).Ortho and Gillham use nano iron to clean water and soil because of the few cost as well as lower toxicity when added to water and soil(Orth, W.S. and Gillham, R.W.1996).Ren. T, and his group studied the preparation of nano-ironfor investigation of Cr(VI) removal capability from synthetic wastewater. Batch experiments revealed that the as-prepared α-Fe_2O_3 nanofibers exhibited excellent Cr(VI) removal performance with a maximum adsorption capacity of 16.17 mg g^{-1}. Furthermore, the adsorption capacity almost kept unchanged after recycling and reusing. The Cr(VI) adsorption process was found to follow the pseudo-second-order kinetics model, and the corresponding thermodynamic parameters ΔG°, ΔH°, and ΔS° at 298 K were calculated to be 78.12 J $mol^{-1}K^{-1}$,(Ren. T, He. P, and Niu .W, 2013).The preparation of nanoparticles of Fe^0(nano-zero valent iron, NZVI) as adsorbent is discussed and the capability of adsorbing methylene blue (MB) is studied. The morphology of the adsorbent was evaluated with transmission electron microscopy. Batch studies were performed to delineate the influence of various experimental parameters such as pH, adsorbent dosage, initial dye concentration, temperature and contact time. Optimum conditions for MB removal were found to be pH 9.5, adsorbent dosage of 0.5 g L^{-1} and equilibrium time of 1 min. The experimental equilibrium data were adjusted by the adsorption isotherms from Langmuir and Freundlich models, and their equilibrium parameters were determined. The adsorption of MB dye by NZVI obeyed both the Freundlich and Langmuir isotherm. The adsorption capacity of NZVI for MB in terms of monolayer adsorption was 208.33 mg g^{-1}(Sohrabi MR 2014)
The aim of this work is to synthesis iron-nano particles by well-established borohydride method. The structure and the prevailing phase were indicated by XRD , AFM , SEM and TEM. Heavy metals adsorption on the surface of prepared nano-partiles was monitored using XPS technique.

2. Experimental methods:

2.1 Synthesis of nZVI

The synthesis of nano iron in this work was done using borohydride reduction of Fe(II) (Wang and Zhang 1997, Wang, et al. 2006a). For this purpose, iron(II) chloride tetrahydrate ($FeCl_2.4H_2O$) and sodium borohydride ($NaBH_4$) were used. In each batch of synthesis, 1.50 g of Fe^{2+} (5.34 g $FeCl_2$.4H2O) was dissolved in a 4/1 (v/v) ethanol/water mixture (24.0 ml ethanol + 6.0 ml deionized water) and stirred on a magnetic stirrer. On the other hand, 1.0 M sodium borohydride solution was prepared; 3.05 g $NaBH_4$ was dissolved in 100.0 ml of deionized water. The borohydride solution was poured in a burette, and then added dropwise to the Fe^{2+} solution, while still stirring on magntic stirrer. Black solid particles immediately appeared after the first drop of sodium borohydride solution. After adding the whole borohydride solution, the mixture was left for further 10 minutes of stirring. The redox reaction can be represnted by :

$$Fe^{2+} + 2BH- + 6H_2O \rightarrow Fe^0 + 2B(OH)_3 + 7H_2 \uparrow \qquad (2-1)$$

Iron nanoparticles were separated from the liquid phase by,vacuum filtration, in which two sheets of blue band Whatman filter papers were used in each filtration process. At this point, the solid particles were washed at least three times with 25.0 ml portions of absolute ethanol to remove water. This washing process is probably the key step of synthesis since it prevents the rapid oxidation of zero-valent iron nanoparticles. The synthesized nanoparticles were finally dried in oven at 500 C overnight

2.1.1: X-Ray Diffraction

X-ray diffractometer (type XRD-6000, Shimadzu) using CuKα radiation was used for the recording of X-Rays with wavelength 1.5406 °A.Powder diffraction was applied to record the pattern of prepared nano iron.

2.1.2: Scanning Electron Microscopy (SEM)

The scanning electron microscopy (SEM) is a powerful technique for analyzing the nano iron. Scanning electron microscopy (SEM) (Type TESCANModelAA8000) at Center of Nanotechnology and Advanced Materials/The Ministry of Science and Technology \ Iraq.

2.1.3: Transmission Electron Microscopy (TEM)

Transmission Electron Microscope is used to observe the internal structure of prepared nano iron prepared. Transmission electron microscopy (TEM) (Type CM10 Philips, Netherlands) at College of Medicine / Al-Nahrain University/Iraq was used in this work.

2.1.4: Atomic Force Microscopy (AFM)

Atomic force microscopy (AFM) is a very high-resolution and one of the foremost tools for imaging, measuring, and manipulating matter at the nanoscale. Atomic Force Microscope (Type Angstrom, Scanning Probe Microscope, Advanced Inc, AA 2000A°, USA). The measurements were conducted at Department of Chemistry/College of Science/University of Baghdad/Iraq.

2.1.5:X-Ray analysisEnergy dispersive(EDX)

XPS, also known as ESCA, is the most widely used surface analysis technique because of its relative simplicity in use and data interpretation. The sample is irradiated with mono-energetic x-rays causing photoelectrons to be emitted from the sample surface. An electron energy analyzer determines the binding energy of the photoelectrons. From the binding energy and intensity of a photoelectron peak, the elemental identity,Made USA and equipped by the Chinese company Sky ray Model 3000 chemical state.at Faculty of science / Al- Ferdowsi University/Iran was used in this work

2:1:6 Atomic absorption spectroscopy(AAS)

Atomic absorption spectroscopy device Malaysia-made model of Phoenix-986 located at Basra University / College of Science.

2.1.7: Effect of Contact Time

In the related experiments, 0.033 g of nZVI sample was addedto 10.0 mL portions of 10^{-4}M metal solutions, and the solutions were mixed for 1 min, 5 min,10min,30min 1 h contact time. After centrifugation and acidification. Followed by analysis byAAS.

2.1.8: Effect of pH,Temperature

The experiments were conducted after adjusting the initial pH's of the mixtures to,(PH= 6, 7, 8) and(T=25,35,50) using NaOH. In each experiment 10.0 mL portions of metal solutions were contacted with 0.033 g nZVI for contact periods of 1 hours. the solutions were acidified and kept in until analysis. The pH of the solutions were also measured at the the end of shaking process After centrifugation and acidification,followed by analysis AAS.

3: RESULTS AND DISCUSSION

Figure (3-1)shows the XRD for nano iron. Scan range was (20.0 – 60.0) deg.The peak at $2\ \theta = 44.6$ which represens 100% intensity, wich indicates the presence of iron α-Fe. As for the peak at $2\ \theta = 38.2$ indicate the presence of iron oxide(Fe_2O_3 or Fe_3O_4). The crystallite sizes were calculated the using Scherrer equation:

$$d = \frac{k\lambda}{\beta \cot\theta} \qquad (3\text{-}1)$$

Where k is a constant about 0.9, λ is the wavelength of the X-rays, β is the full width of diffraction peakat half maximum intensity and θ is the Bragg angle. The calculated crystallite sizes were found to be inthe range of 35–44 nm.

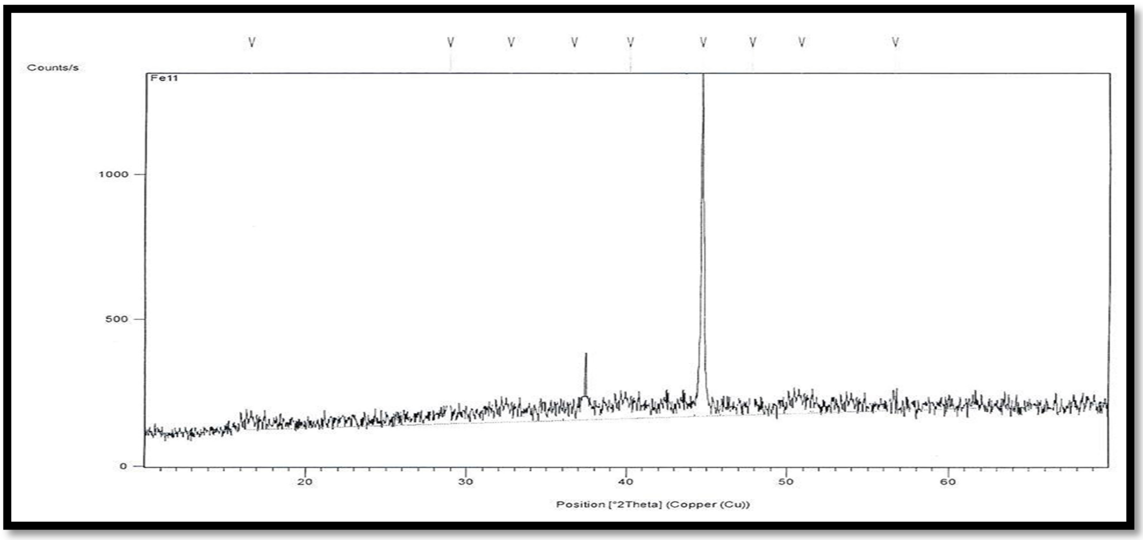

Figure 3.1 XRD patterns of nano iron

Figure (3-2a) shows Atomic force microscope pictures the diameters of prepared nano iron in range of (20– 110 nm) andFigure(3-2b) average diameter was 67.5 nm

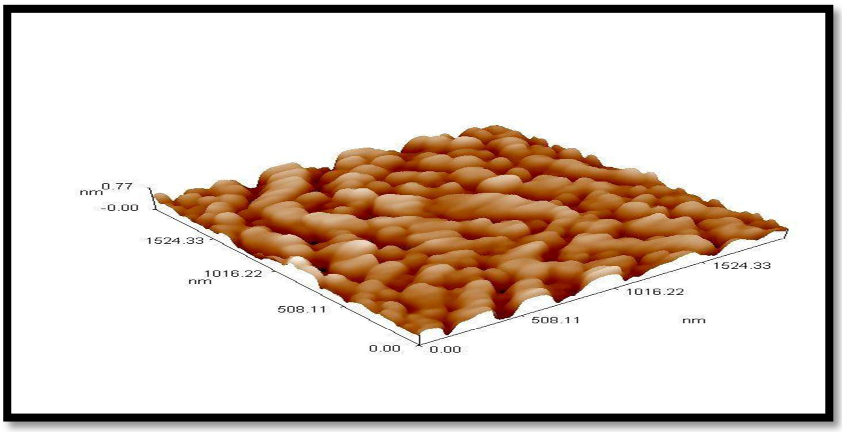

Figure (3-2a): partied size distribution

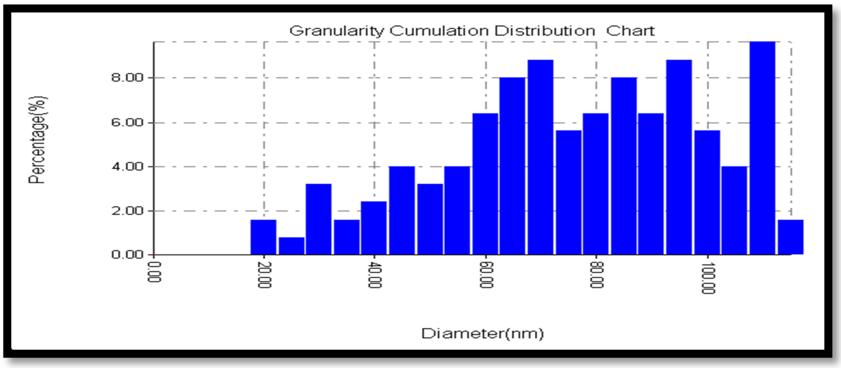

Figure (3-2b): Atomic Force Microscopy (AFM) picture

3-1-3 Scanning Electron Microscopy (SEM):

Typical SEM images of nZVI, shown in Figure (3.3), indicate the nanoparticles formed which hase a chain-like aggregates that can reach to several micrometers in length but are less than 90 nm in diameter. Aggregation of nanoparticles is reported to be caused by the large surface area and magnetic dipole-dipole interactions of the individual particles (Li, et al. 2006),where size of the aggregates was determined through the bottom of the image scale.

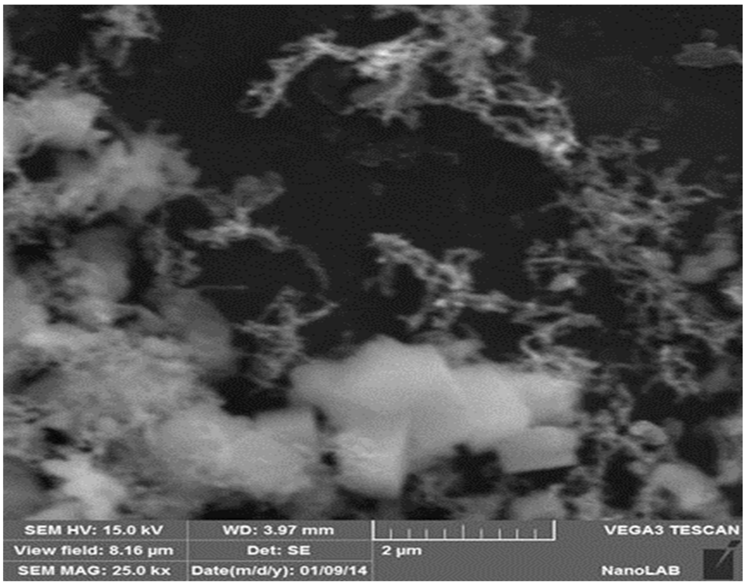

Figure (3-3): SEM image nano iron particles

3-1-4 Transmission Electron Microscopy (TEM):

The internal structure of sample of nano iron prepared was identified by transmission electron microscope (TEM). Figure (3-4)The nano iron particles appear in the characteristic chain-like structure, with the size of individual particles ranging within 20-66 nm.

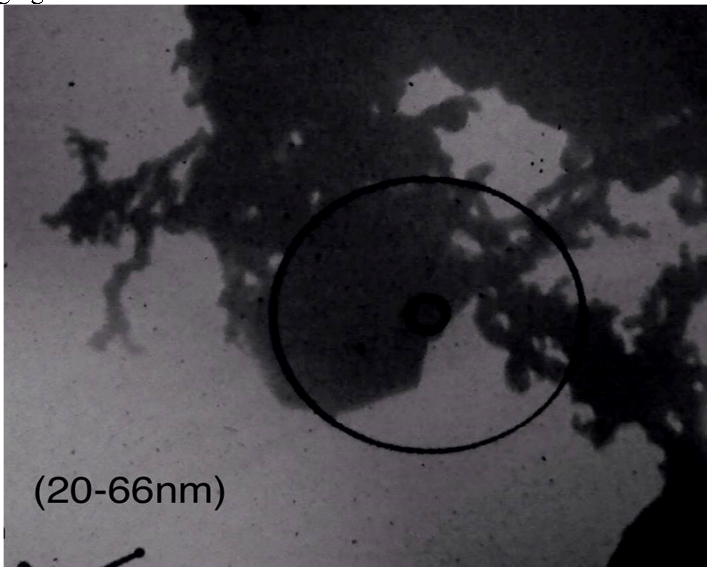

Figure (3-4): TEM image of nano iron particles

3-2 ADSORPTION:

The pH of the aqueous solution is an important factor to affect the (Ni,Cd,Cr) adsorption process through changing the surface charge of an adsorbent and the ionization behavior of adsorbent and (Ni,Cd,Cr) . The influences of the solution pH on(Ni,Cd,Cr) removal % and adsorption capacity by naon iron are shown in Figures (3-5),(3-6),(3-7)

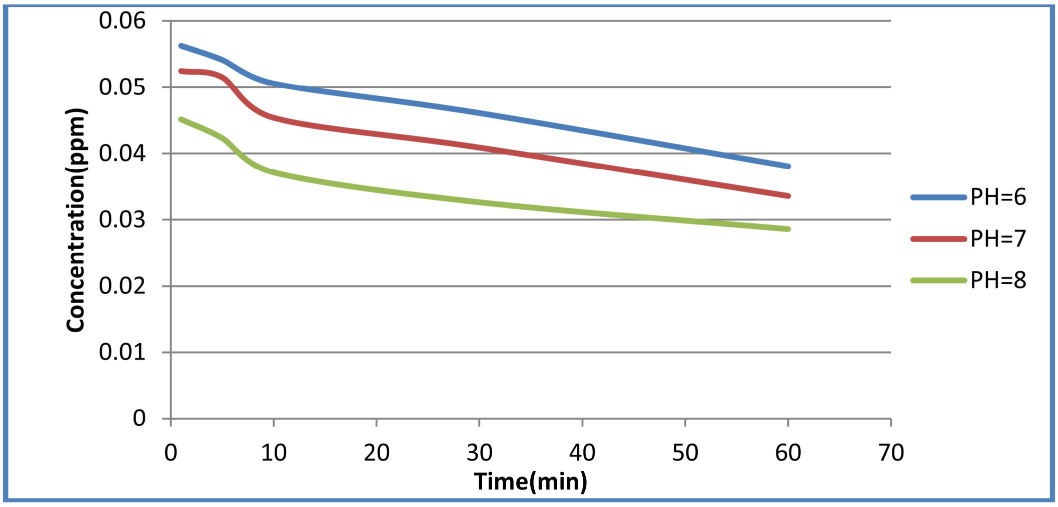

Figure 3.5: effect of PH on Cd adsorption

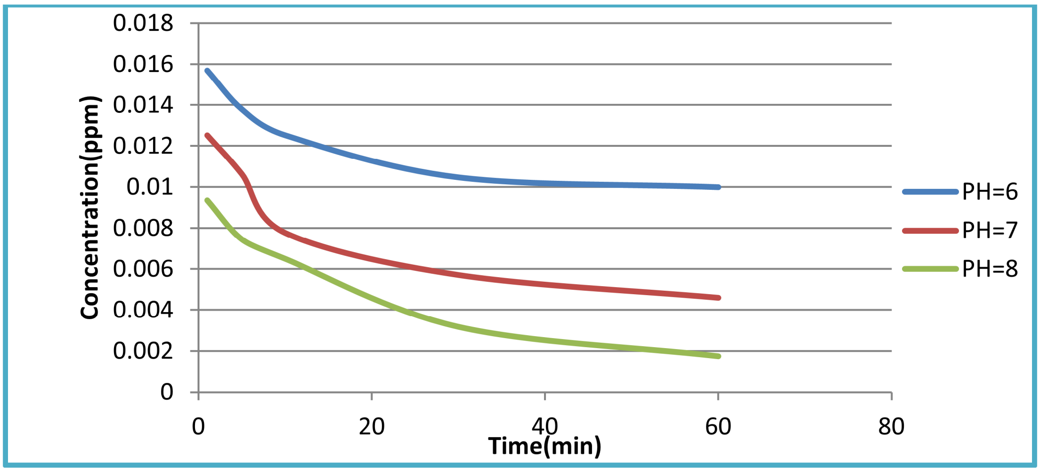

Figure 3.6:effect of PH on Cr adsorption

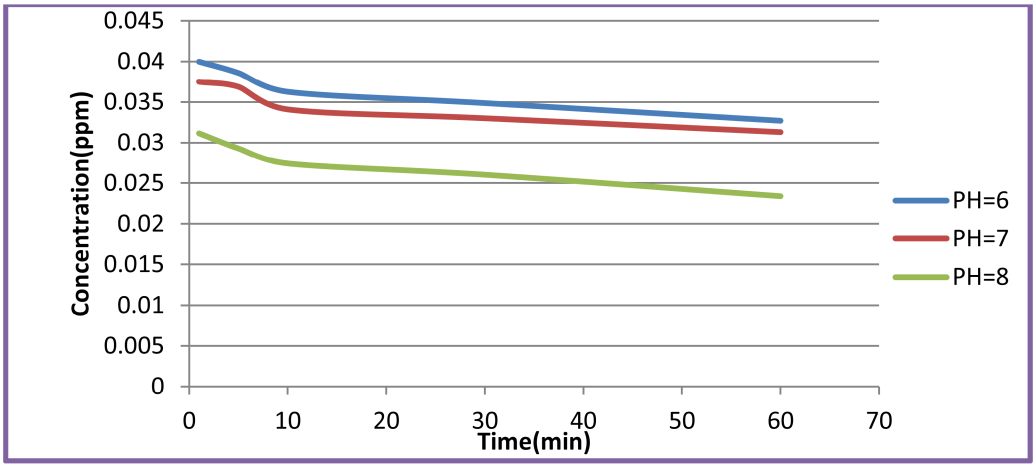

Figure 3.7:effect of PH on Ni adsorption

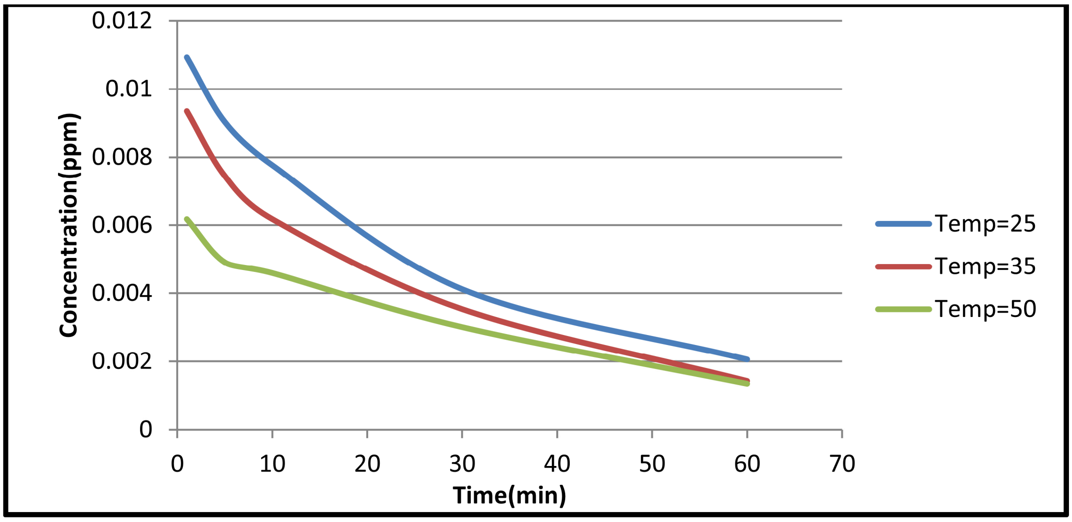

Figure 3.8:effect of temperature on Cd adsorption

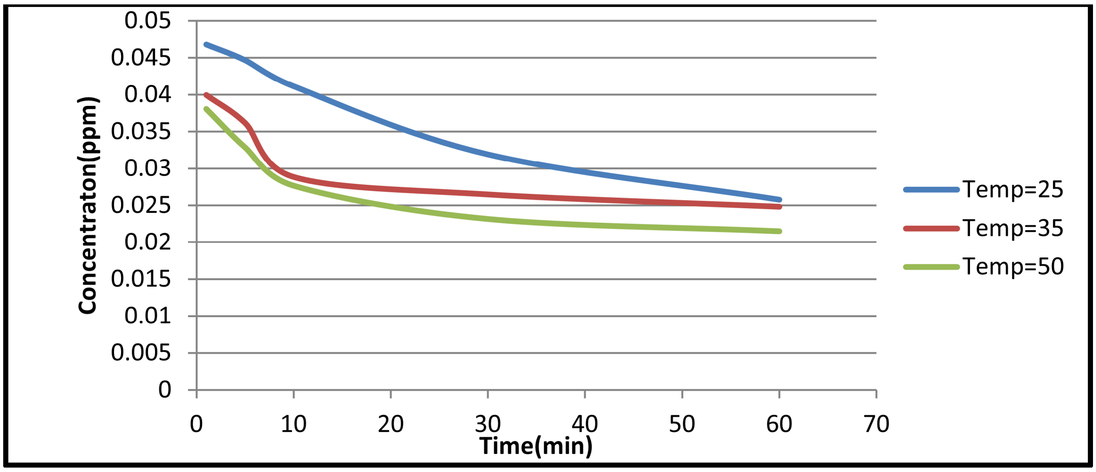

Figure 3.9:effect of temperature on Cr adsorption

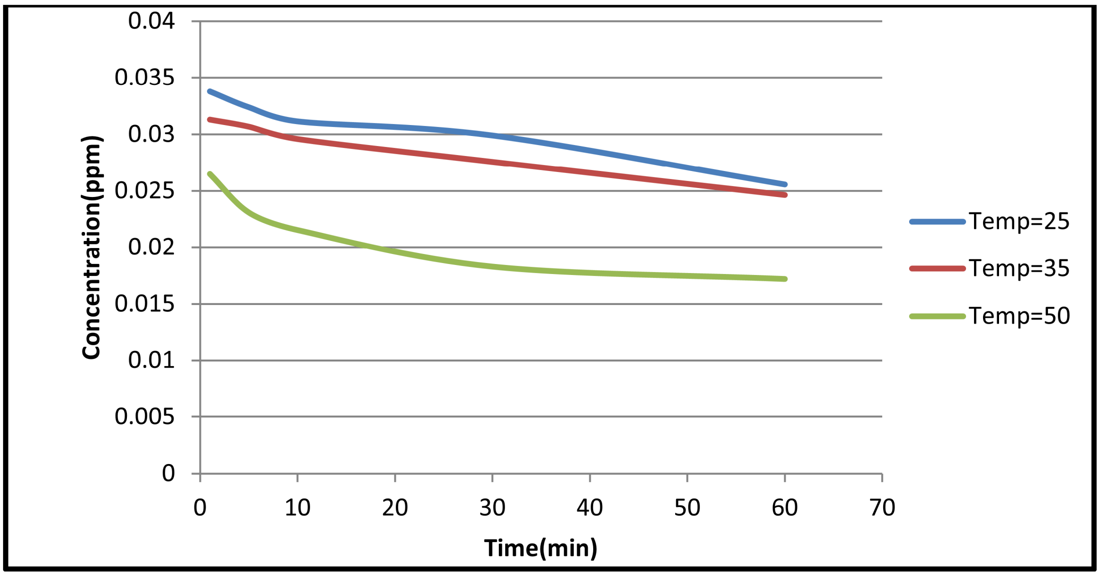

Figure 3.10:effect of temperature on Ni adsorption

3.3 Adsorption Isotherms:

In this study, isotherm Langmuir models.were studied to describe the equilibrium characteristics of adsorption.

Langmuir isotherm was excluded because the lack of linearity of the results ther for Freundlich isotherm were adopted. The plot of log Ce versus log q Figure (3-11), Cd, (3-12)Cr and (3-13) Ni the linear regression correlation (R^2) of Langmuir isotherm .

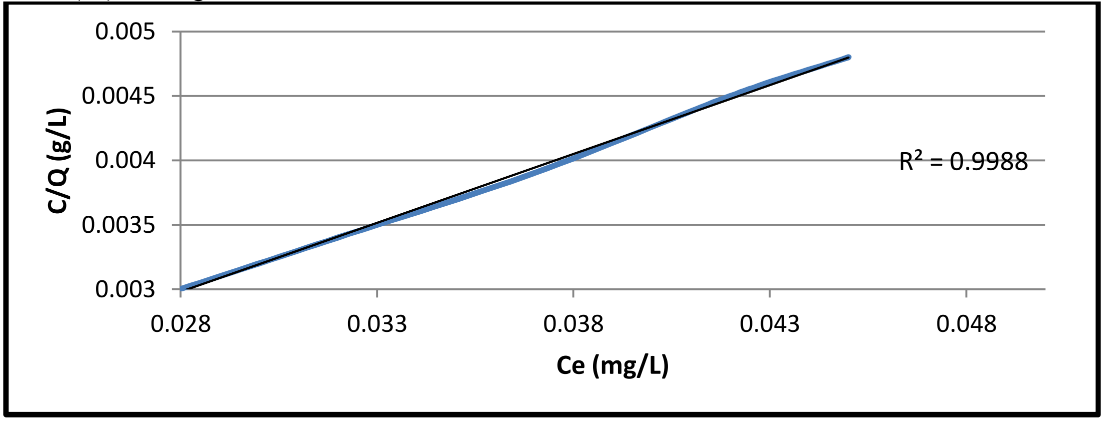

Figure (3-11): Langmuir isotherm of Cd

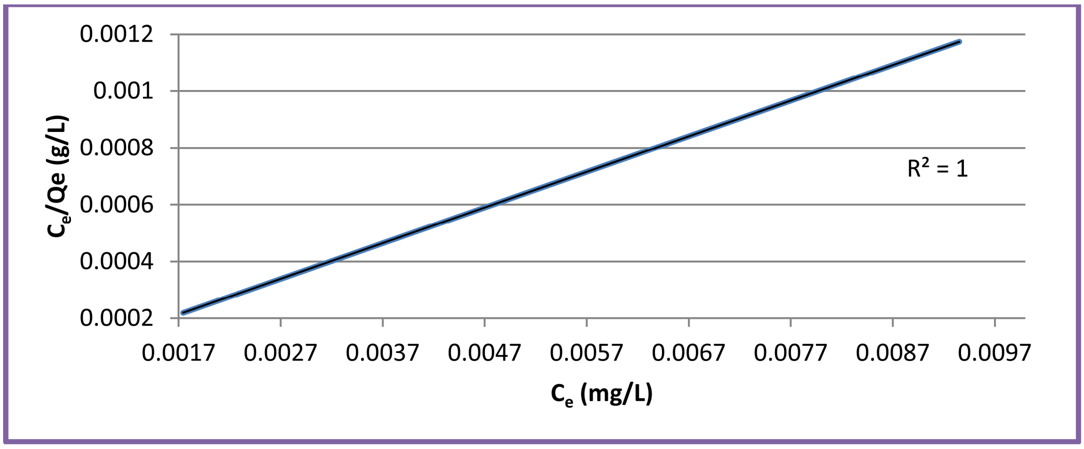

Figure (3-12): Langmuir isotherm of Cr

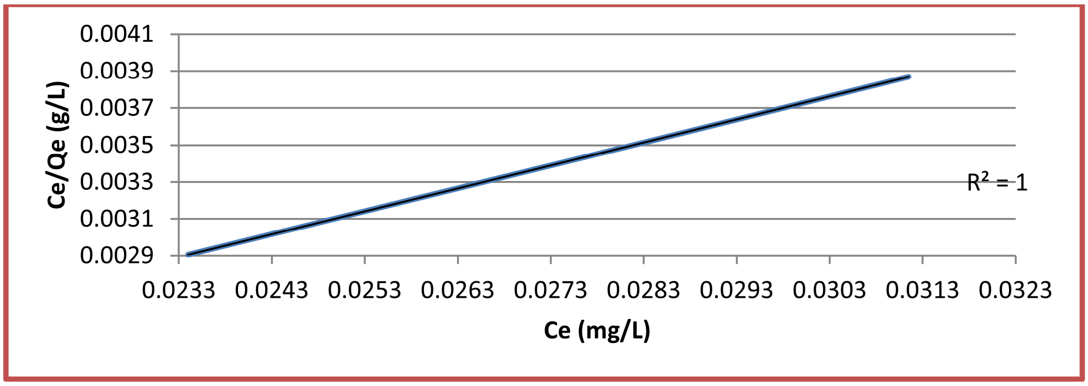

Figure (3-13): Langmuir isotherm of Ni

Table 1: fraundlich constants for Cd,Cr,Ni

constants	**PH=7 , T=25**		
	Cr	**Cd**	**Ni**
R^2	**1**	**0.99**	**1**
b_L	**0.2**	**0.8**	**0.6**
q_{max}	**0.02**	**0.08**	**0.08**

3.4 Thermodynamic parameters:

Through previous results have shown the effect of temperature on the rate of adsorption.was taken identical for all temperatures of study. thus a plot $\log X_m$ against 1/T gives a straight line with slope equal $-\Delta H/2.303R$.

$$\log x_m = \frac{-\Delta H}{2.303RT} + K \qquad (3\text{-}3)$$

Where X_m is the maximum uptake of adsorption at a ertain value of the equilibrium concentration(C_e)

Can also calculate the value of ΔG through the following relationship

$$\Delta G = -RT\, In(\frac{Qe}{Ce}) \qquad (3\text{-}4)$$

Gibbs application equation are getting ΔS

$$\Delta G = \Delta H - T\Delta S \qquad (3\text{-}5)$$

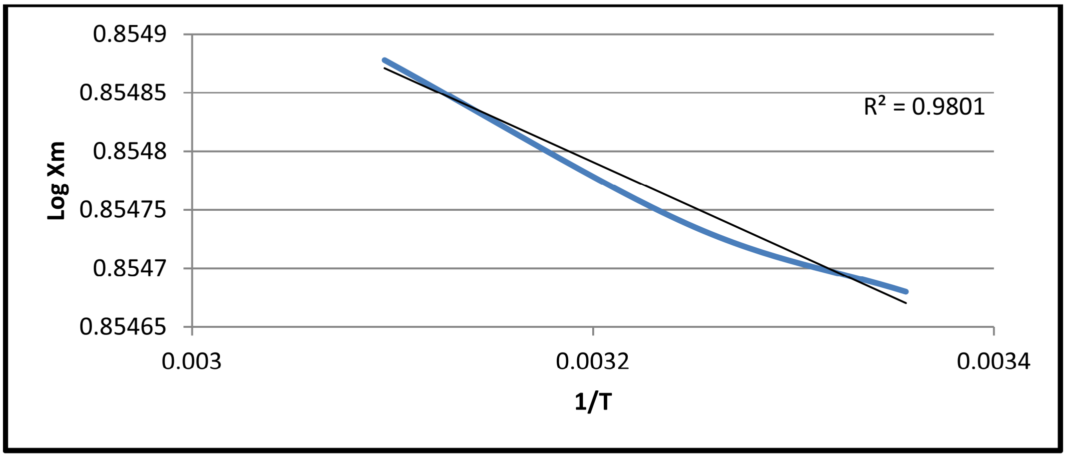

Figure (3-14): plot $\log X_m$ against 1/T Cd^{+2}

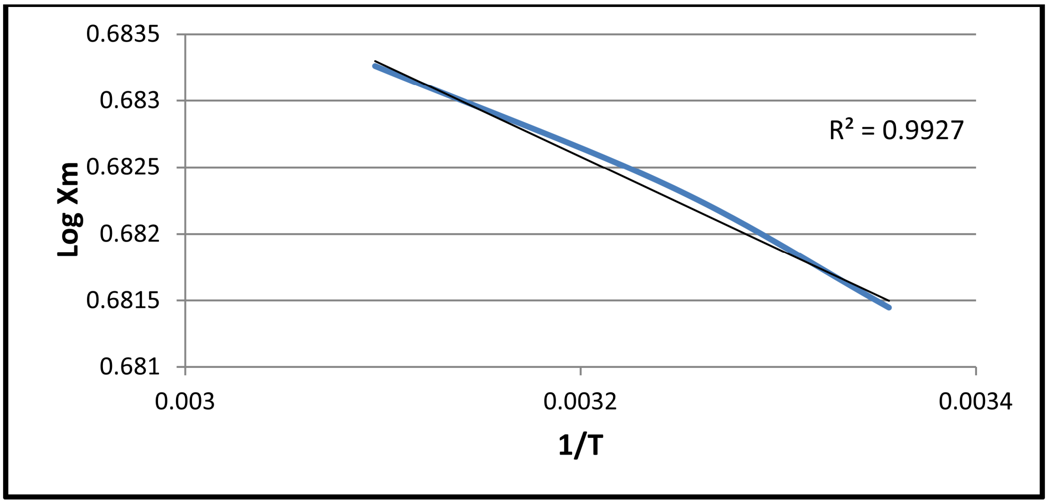

Figure (3-15): plot $\log X_m$ against 1/TCr^{+2}

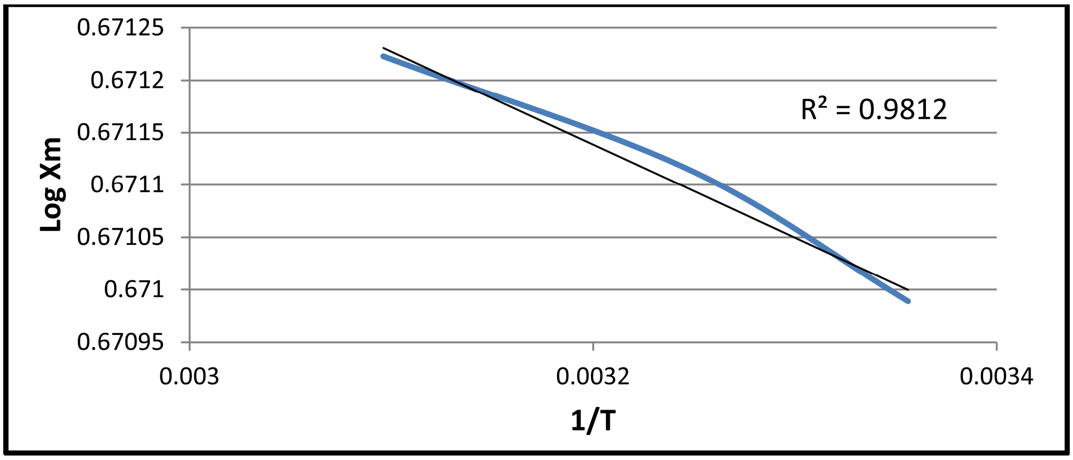

Figure (3-16): plot logX_m against 1/TCr^{+2}

Table 2:thermodynamic parameters of adsorption process ofCd,Cr ,Ni on the adsorbents 25C^0

Element	) H(KJ.mol^{-1}Δ	) G (KJ.mol $^{-1}$Δ	S (J.mol^{-1}k^{-1}) Δ	K
Cd	**11.6**	**-12.6**	**0.08**	**0.85**
Cr	**22.8**	**-15.5**	**0.13**	**0.71**
Ni	**16.6**	**-12.9**	**0.098**	**0.67**

3.5:X-Ray analysisEnergy dispersive(EDX)

Figure (3.17b),(3.18b),(3.19b)shows the high resolution scanning electron microscope image of Fe after adsorption of Cr,Ni,Cd ions. the particle are showing an increased agglomeration and surface of the particles of Fe after adsorption of the Cr,Cd,Ni ions is depicted in From the EDX spectrum, it is observed that there is major component of Fe and O due to Fe3O4 and presence of Cd,Cr,Ni adsorbed on it appearancepeakoxygen and the presence of oxygen at peaks (640,60ev)indication of the presence of iron oxides, and this is due to exposure to water and oxygen as the infrastructure is FeO while core contains iron oxide (Fe2O3, Fe3O4, FeOOH(Zhang W and Elliot2006) by t mechanisem (Li XQ 2006):

Mechanical removal ofCd^{+2}
$Cd^{+2} + FeO \leftrightarrow FeOCd^{+} + H^{+}$ (3-6)
$Cd^{+2} + 2(FeOH) \leftrightarrow (FeO)_2\,Cd + 2H^{+}$ (3-7)
$Cd^{+2} + FeOH + H_2O \leftrightarrow FeCdOH + 2H^{+}$ (3-8)
Mechanical removal ofCr^{+2}
$Cr^{+2} + FeO \leftrightarrow FeOCr^{+} + H^{+}$ (3-9)
$Cr^{+2} + 2(FeOH) \leftrightarrow (FeO)_2\,Cr + 2H^{+}$ (3-10)
$Cr^{+2} + FeOH + H_2O \leftrightarrow FeCrOH + 2H^{+}$ (3-11)
Mechanical removal ofNi^{+2}
$Ni^{+2} + FeO \leftrightarrow FeONi^{+} + H^{+}$ (3-12)
$Ni^{+2} + 2(FeOH) \leftrightarrow (FeO)_2\,Ni + 2H^{+}$ (3-13)
$Ni^{+2} + FeOH + H_2O \leftrightarrow FeNiOH + 2H^{+}$ (3-14)

Through previous results for your EDX can be seen removing cadmium, chromium and nickel are by adsorption to iron through E0 where we note that whenever it was the biggest iron contaminant, the removal process is the adsorption process but if the approach or smaller than the iron, the process is the absorption and can be seen as the E0 Steel (-0.44) while the E0 value of pollutants are (Cd = -0.40, Cr = 1.36, Ni = -0.23 (Ponder SM 2000, Lien HL 2007). Table Standard redox potentials (E^0) in aqueous solution at 25 C(Bard AJ, Parsons R,and Jordan J1985).Figure (3.17a),(3.18a),(3.19a) Note the size of the largest minutes compared with the images then diagnosed by sem before adsorption process and this indicates adsorption of heavy metals by iron nano.

Aqueous solution	Half reactions	E^0 (V)
Ni	$Ni^{2+} + 2e^{-} \longleftrightarrow Ni$	-0.25
Cd	$Cd^{2+} + 2e^{-} \longleftrightarrow Cd$	-0.40
Cr	$Cr^{3+} + e^{-} \longleftrightarrow Cr^{2+}$	0.41
Fe	$Fe^{2+} + 2e^{-} \longleftrightarrow Fe$	-0.44

Table 3:EDX for Cd

Element	Weight%	Atomic%
O K	40.60	70.49
S K	2.31	2.00
Fe K	53.54	26.63
Cd L	3.55	0.88
O K	40.60	70.49

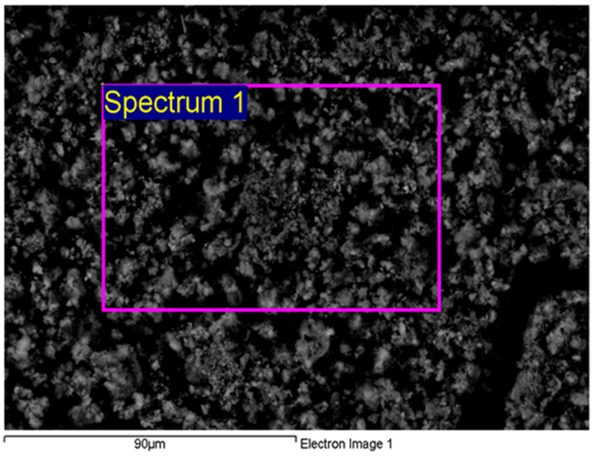

Figure 3.17a:EDX image Cd adsorped on Fe

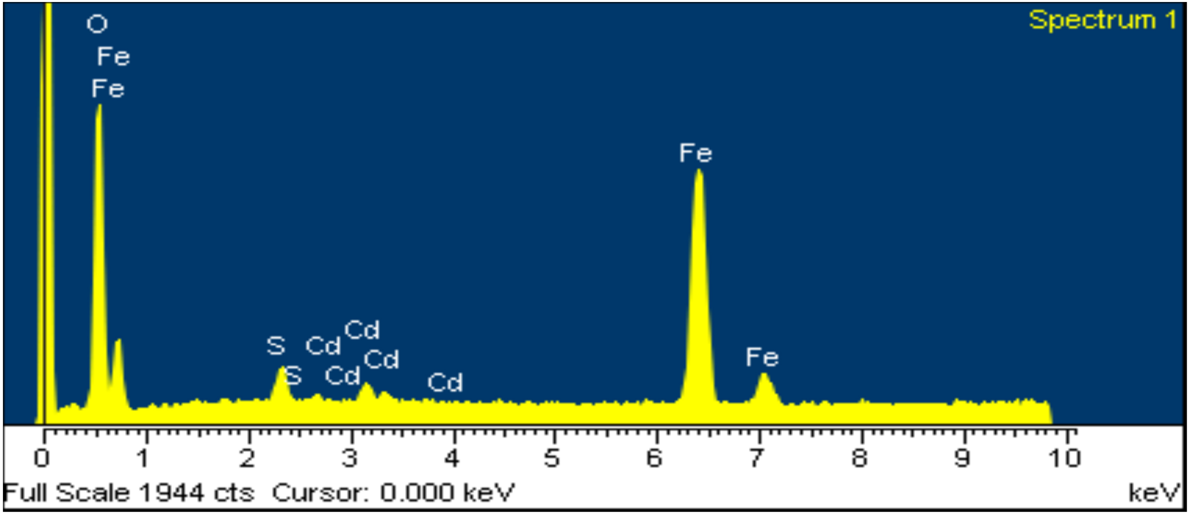

Figure 3.17.b. (EDX) analysis of nano iron particles the adsorption of Cd^{+2} ions

Table4: EDX for Cr

Element	Weight%	Atomic%
O K	38.34	67.13
Al K	0.64	0.67
Si K	0.68	0.68
S K	3.01	2.63
Cr K	3.64	1.96
Fe K	53.68	26.93

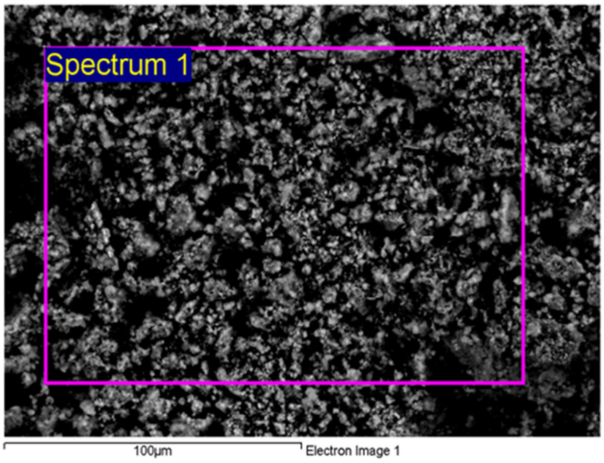

Figure 3.18a:EDX image Cr adsorped on Fe

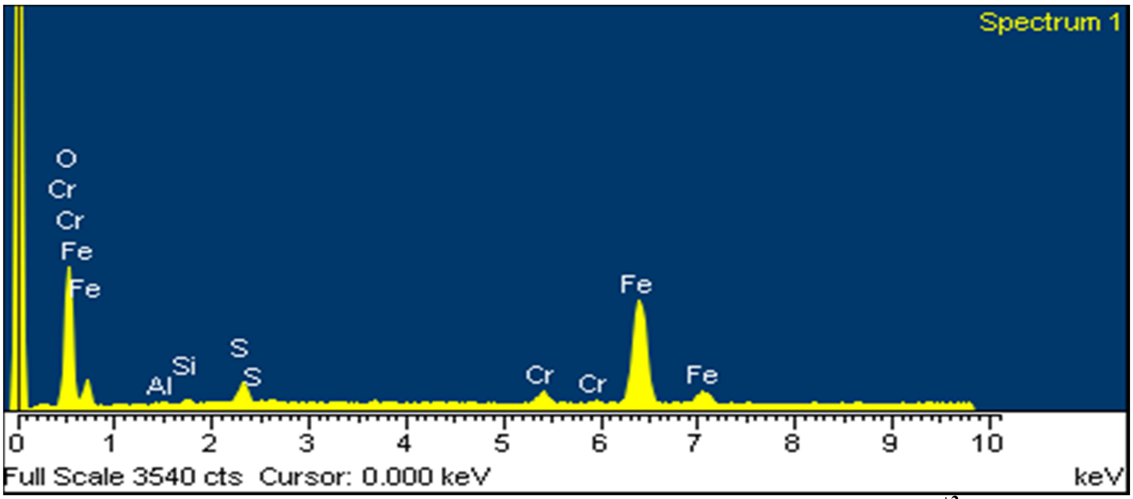

Figure 3.18b: (EDX) analysis of nano iron particles the adsorption of Cr^{+2} ions

Table 5: EDX for Ni

Element	Weight%	Atomic%
O K	41.84	70.60
Al K	0.70	0.70
S K	2.67	2.25
Fe K	53.05	25.65
Ni K	1.75	0.80

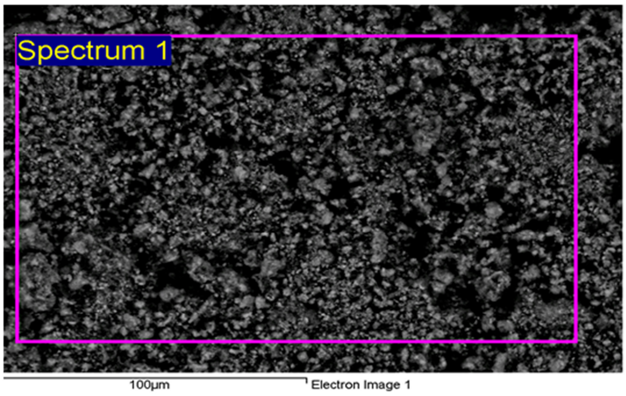

Figure 3.19.a: EDX image Ni adsorped on Fe

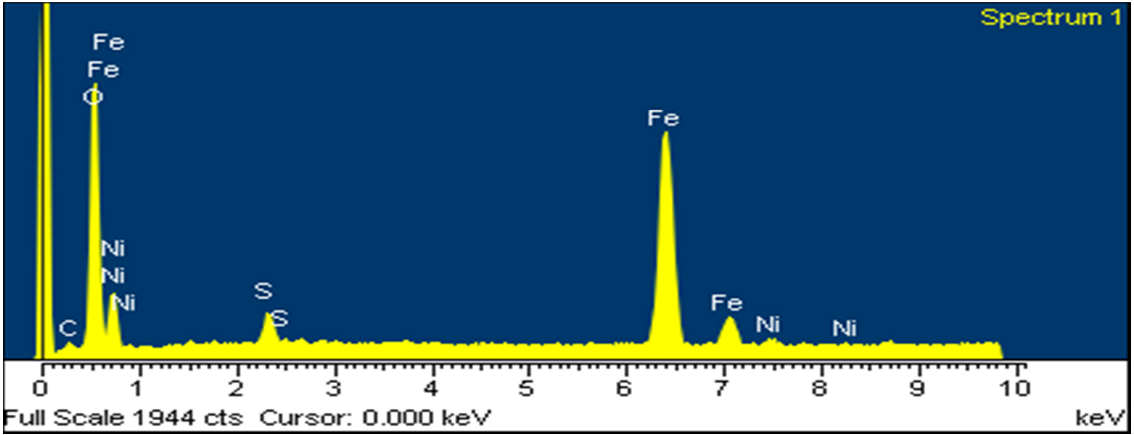

Figure 3.19b: (EDX) analysis of nano iron particles the adsorption of Ni^{+2} ions

Conclusions

Nano zero valent iron can be used as an effective adsorbent for removing Cd,Ni,Cr from contaminated water sources. Increasing the temperature increased the cadmium adsorption rate but the maximum adsorption capacity was similar. The pseudo second order kinetic model accurately described the adsorption kinetics. The adsorption mechanism was found to be chemisorption and the rate-limiting step was mainly surface adsorption. The Langmuirisotherm showed a better fit than the Freundlich isotherm, thus, indicating the applicability of monolayer coverage of cadmium on nZVI surface. The equilibrium data were also well described by the Temkin equation further supporting cadmium adsorption on nZVI as a chemisorption process. Thermodynamic analysis showed that the adsorption process was endothermic and spontaneous in nature. EDX analysis confirmed that Cd2+ was adsorbed onto the nZVI particles. Results from this study suggest that nZVI is a very effective adsorbent for cadmium, as anticipated.

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