The environmental application over nickel supported nano-structured silica from agricultural biomass

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Abstract:

The objective of this study is the removal of some environmental hazards from aqueous solutions using six nickel on two supports prepared by impregnation and sol-gel methods. The first support was rice husk ash (SiR) and the other was silica-gel SiG by different loading weight percent of nickel 4.80, 9.60, 14.80, 4.90, 9.80 and 14.70% respectively, which were produced, Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII, respectively. The solids were described by TGA, DTA, DSC, XRD, TEM and BET measurements.The surface analyses show that for the bare SiR support is a common amorphous structure and in Ni/SiR catalyst precursors after calcination at 773 K, nickel is presented as NiO species. TEM images depict that nickel crystallites are spherical in shape, its size is in nano-scale and much lower than 14 nm and distributed uniformly. The adsorption of hazard material likeCr^{+6} is maximal at the lowest values of pH and increases with temperature for the prepared solids. Optimum conditions for Cr^{+6} removal were found to be pH =3.00, adsorbent dosage 0.05 g/L of solution and equilibrium time 2.00 h. The experimental data were analyzed using Freundlich, Langmuir and Dubinin–Radushkevich (D–R) isotherm models. It was found that Freundlich and D–R models fitted well. The value of the adsorption energy calculated using D–R isotherm, was 1.20±0.10 kJ /mol for Ni/SiR and Ni/SiG solids and it indicated that the adsorption process was physical in nature. The Ni/SiG solids shows higher capacity for adsorption of Cr(VI) than Ni/SiR solids.

Key words: Rice husk ash silica ; NiO catalyst ; Nano silica support ; Impregnation method ; Sol-gel method ; Chromium hexavalent

1. Introduction

The need for cost–effective removal of dyes and pigments from wastewater has led to a proliferation of research into the use of low cost agro-waste adsorbent for dye removal. Dye production industries and many other industries which use pigments and dyes are increasing globally by the day with advancement in technology. Presence of pigments in wastewater constitute environmental hazard because pigments are difficult to degrade.

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Pigments containing chromium, as environmental contaminant, can enter into the ecosystem fromelectroplating, chromate preparation, metal finishing, leather tanning, atomic power plants، mining, cooling towers of heavy industry, metallurgy operations etc(Khezami & Capart, 2005). Strong exposure of Cr^{+6} causes cancer in the digestivetract and lungs and may cause epigastria, nausea, vomiting, severe diarrhea and hemorrhage(Huang & Wu, 1977). It is therefore, essential toremove$Cr^{+6}$from wastewater before disposal. Numerous treatment methods such as chemical reduction, photo-catalytic reduction, ion exchange, membrane separations, chemical precipitation, electrochemical precipitation, adsorption and biosorption have been tested for chromium laden wastewater remediation in yester years(Faust & Aly, 2013; Kadirvelu et al.,2001).Most of these technologies are quite satisfactory in terms of purging chromium from wastewater but many of these produce sludge containing important concentrations of toxic ions like Cr, Cu, Cd, Pbetc whose final disposal is still a problem. The commercially available activated carbon in powder or granular form is effective for the removal of various pigments, dyes and heavy metal ions(Bansal, et al., 2009). However, due to prohibitive cost their use is limited in developing countries like Egypt. In recent years, several waste like rice husk ash (RHA) has been tested for their heavy metals and pigments removal efficiency from synthetic wastewaters. Several authors have reported the efficiency of RHA as an adsorbent for the individual removal of Ni^{+2}, $Cd^{+2}$and Zn^{+2} from aqueous solutions(Kadirvelu et al., 2001). The present investigation is devoted to study the removal of pigment containing, Cr^{+6} from wastewaters by using six prepared solids of Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII. The effects of pH, adsorbent dose, temperature, concentration of metal ions and contact time have been investigated. The data obtained may be useful for environmental hazard removal.

2.Materials and Methods

This study dealing with two groups of solids are I. prepared from RHA by impregnation method ,and II- prepared from TEOS by sol-gel method. In the first group ,Rice husk, was received and isolated from a rice mill that grow in Kom Hamada, Al Buhayrah, Egypt. The rice husk was washed thoroughly with distillated water in order to remove adhering soil and then it was dried in an air oven at 373K for 24h. The dried rice husk was refluxed with 3.00 N HNO₃ in a glass round-bottomed flask at its boiling point 373 K for 1.00 h. The mixture was then filtered and washed repeatedly with warm distilled water until the filtrate was free from acid. The material was then dried at 373 K for 24.00 h and then heated in an air furnace at 1173 K for 1.00 h to remove carbon. The white rice husk ash was obtained with amorphous silica content higher than 99.00%. Such ash was used as a support for catalyst preparation and designated as SiR. The Ni catalysts supported on SiR were prepared by the impregnation technique .A solution of nickel nitrate of known concentrations 4.80, 9.60 and 14.80% was allowed to pass drop wise over SiRto produce Ni/SiRI, Ni/SiRII and Ni/SiRIII with constant stirring see Table(1). The products obtained were dried in an electric oven at 420K for 4.00

hour. The solid was crushed in Pyrex mortar. The desired diameter of grain size (0.25-0.5mm) was chosen for adsorption measurements by using appropriate sieves and calcining at 773 K for 4.00h.The other catalysts were prepared by sol-gel method in which SiO_2 sol was obtained by hydrolyzing 31.91g of TEOS in mixture of 21.26g of THF , 6.74 g of H_2O and 0.75g of 2.00M of HCl under magnetic stirring at 65-70°C for 3h . The SiO_2 sol was then mixed with a solution of nickel nitrate with 4.90, 9.80 and 14.70% concentration to produce Ni/SiGI, Ni/SiGII and Ni/SiGIII respectively, was allowed to pass drop wise with constant stirring for 24.00h. Finally , the cross linking agent $Si(OC_2H_5)_4$ was added to the system to form a transparent binary gel .The gel obtained was dried at room temperature for 2.00 days, ground into fine powder and then heated at 323K for 5.00 h and calcining at 773K for 4h. Samples of waste water of $Cr^{+6}$was received from Qaha, factory for chemical industries,Qalyoubia , Cairo, Egypt, and pH of thesolutions was adjusted using 0.01 M NaOH/0.01 M HCl using pHmeter (Model pHep, Hanna Instruments, calibrated with buffers. The Cr^{+6} concentration was determined byAtomic Absorption spectrophotometer(Shimadzu 6300, Japan).Batch experiments were carried out at different pH (3.00–9.00), adsorbent dose (0.01–0.05g /l) and stirring speed (180 rpm) for a contacttime of (30-180) min. For each batch experiment, 10 ml solution of waste water was used.

3. Results and Discussion

3.1.Effect of pH

ThepH of the medium has a significant effect on the sorption of metal ions on different adsorbents. This is partly due to the fact that the hydrogen ion itself is a strong competing adsorbate and partly due to the chemical speciation of metal ions under the influence of the solutionpH. At acidic pH, the predominant species of Cr are $Cr_2O_7^{-2}$, $HCrO_4^{-1}$,H_2CrO_4, $Cr_3O_{10}^{-2}$ and $Cr_4O_{13}^{-2}$. The adsorption of metal ions depends upon the nature of the adsorbent surface and the species distribution of the metal ions in the aqueous solution.

Table (1) : An overview of supports used and their pretreatment conditions.

| Samples	Silica Source	Chemical Composition of Ni %	True chemical Composition by A.A. of Ni %	Specific surface area , $m^2.g^{-1}$	Method of Preparation	Calcinations Temp., K
Ni/SiRI	RHA	5.00	4.80	48.70	Impregnation	773
Ni/SiRII		10.00	9.60	70.35		
Ni/SiRIII		15.00	14.80	125.00		
Ni/SiGI	TEOS	5.00	4.90	335.00	Sol. gel.	773
Ni/SiGII		10.00	9.80	274.00		
Ni/SiGIII		15.00	14.70	202.00		

The effect of pH on the removal of Cr^{+6} is investigate by testing three values of pH = 3.00, 6.00 and 9.00 at room temperature for the tested wastewater (10.00 mg/l) over 0.01 g/l for all the prepared solids. The contact time has been fixed to 1.00 h for all the experiments. The results presented in Fig.(1) indicate that Cr^{+6} removal is considerably affected by the pH. The maximum adsorption of Cr^{+6}pigmentwas observed at pH equal 3.00 for all prepared solids. At initial pH of 3.00, the adsorbent surfaces might be highly protonated which favor the uptake Cr^{+6} in the predominant anionic form(Dakikyet al., 2002; Raoet al., 1992). With increase in pH, the degree of protonation of the surface reduces and hence adsorption is decreased. Furthermore, as pH increases, there is competition between OH^- and chromate ions. Hence, adsorption of Cr^{+6} on the adsorbent was not significant at greater pH values due to dual competition of both the anions($CrO_4^{2-}$and OH^-) to be adsorbed on the surface of the adsorbent of which OH^- predominant. Maximum adsorption occurs at pH 3.00 and hence it was taken as the optimal value for further adsorption studies for all solids. Generally for all the prepared Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII solids by increasing of Ni content, the removal of the pigments increase.
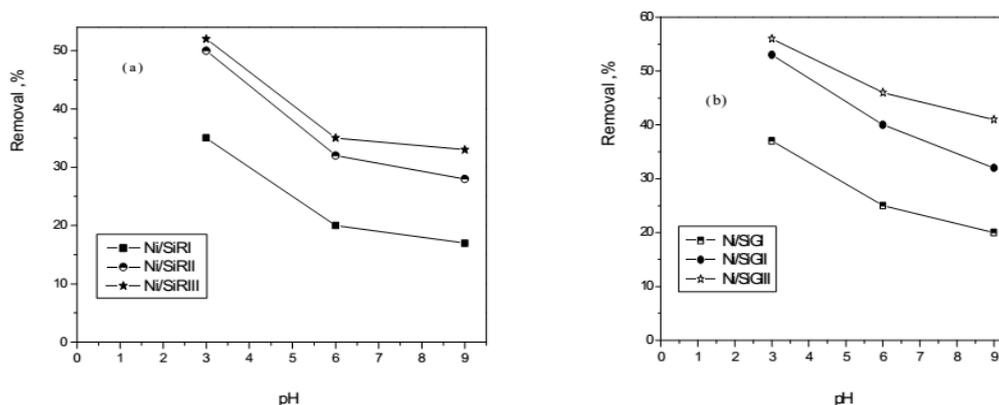
Fig.(1) : *Effect of pH on the adsorption of Cr(IV) from waste water onto a)Ni/SiR and b)Ni /SiG solids.*

3.2 Effect of adsorbent dose

The removal of metal ions is a function of adsorbent dosage, so the efficiency of the prepared solids was evaluated at different adsorbent doses for the percent removal of chromium. The experiments were conducted at constant initial concentration=10.00 mg/l, contact time =1.00 h, pH= 3.00, at room temperature and stirring speed =180 rpm with varying adsorbent doses 0.01–0.05 g/ l. Measurement of percentage Cr^{+6} removal as a function of time at different doses indicates that removal of Cr^{+6} increased with increasing adsorbent dose (Fig.(2)). Increase in Cr^{+6}removal with adsorbent dose can be attributed to increased S_{BET} and the availability of more adsorption sites (Kadirvelu et al., 2000)for all the prepared solids.
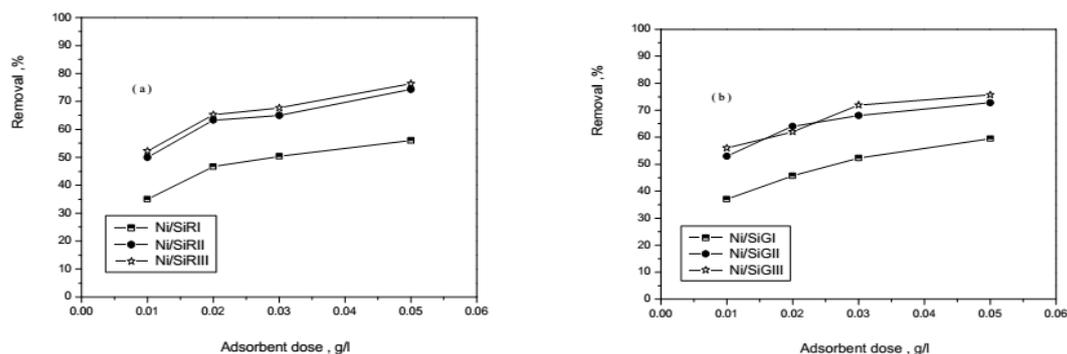
Fig.(2) : *Effect of adsorbent dose on the adsorption of Cr(IV) from waste water onto*
a) Ni/SiR andb) Ni /SiG solids.

3.3. Effect of contact time

The effect of changing the contact time on adsorption, while keeping the adsorbent dose= 0.01 g/l, pH=3.00 and stirring speed =180 rpm constant at room temperature illustrated in Fig (3).The removal of Cr^{+6} has increased with time and attains a maximum value at about 120 min and reached to 60.43, 75.97, 77.66, 62.77, 78.52 and 81.67 % for Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII solids respectively and thereafter, it remains almost constant. This may be due to the fact that as the pigment solution-adsorbent system is being agitated at longer time, more of the molecules or atoms of the pigment tend to accumulate on the surface of the adsorbent until equilibrium is reached.
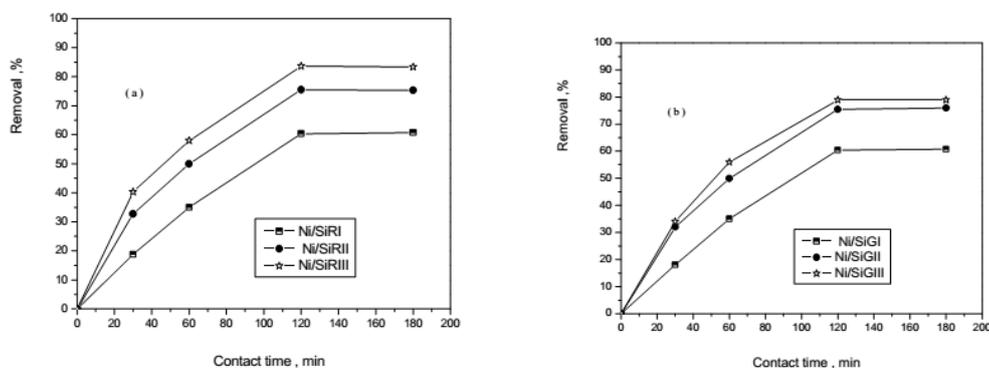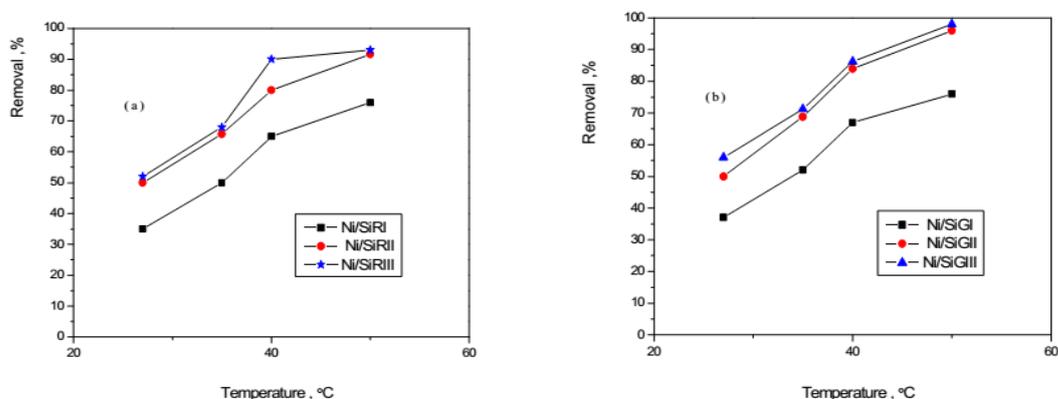

Fig.(3) : *Effect of contact time on the adsorption of Cr(IV) from waste water onto*
a) Ni/SiR and b) Ni /SiG solids.

3.4.Effect of temperature

An increase in temperature is known to increase the diffusion rate of the adsorbate molecules across the external boundary layer and within the pores. This could be the result of decreasing the viscosity of the solution. Moreover, changing the temperature will modify the equilibrium capacity of the solids for a particular adsorbate(Ho et al., 2001). The effect of temperature is investigated from adsorption tests carried out at three constant temperatures:

300, 308 and 313K for 1.00 h, the adsorbent dose= 0.01 g /l, pH =3.00 and stirring speed =180 rpm. The removal of Cr^{+6} has increased with temperature and attains a maximum value at about 323K and reached to 76.06, 91.69, 93.87, 76.06, 95.93 and 98.00% for Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII solids respectively

Fig.(4) : *Effect of temperature on the adsorption of Cr(IV) from waste water onto a) Ni/SiR and b) Ni /SiG solids.*

3.5. Effect of initial concentration

At time 120 min, initial metal concentration10.00, 20.00, 50.00 mg/l , pH= 3.00, stirring speed=180 rpm at room temperature and adsorbent weight 0.01g. Table(2) showed the equilibrium absorbance (q_e) and % of removal of pigment on the prepared adsorbents. From Table (2), it is evident that the percent of heavy metals ions removal decreased when C_i increased although the actual amount of metal ions adsorbed per unit mass of adsorbents similar observation was reported by Kumar et al (2010).The results shows that Cr^{+6}pigment removal is not upto the permissible disposal limit as prescribed by National Standards. But as the removal of Cr^{+6}pigment is dependent on pH, C_i, temperature and adsorbent dose, so when C_i is low, adsorbent dose is higher and pH is optimum, then the permissible levels of Cr^{+6} pigment can be achieved in the effluents. At low concentration, the ratio of available surface to the initial Cr^{+6}concentration is larger, so the removal is higher. However, for higher concentrations this ratio is low, hence the removal, percentage is also lesser. Increasing metal ion concentration decreased % of Cr^{+6} removal And reached to 30.98, 50.87,55.77,39.98, 57.56 and 60.00 for Ni/SiRI, Ni/SiRII, Ni/SiRIII, Ni/SiGI, Ni/SiGII and Ni/SiGIII. This could be attributed to increased rate of mass transfer due to increased concentration of driving force.

3.6. Adsorption isotherms

The Freundlich, Langmuir and Dubinin−Radushkevich (D−R) models have been employed as adsorption isotherm models. These isotherms relate the amount of solute adsorbed at equilibrium per unit weight of adsorbent to the adsorbate concentration at equilibrium, C_e, mg/l. The Freundlich isotherm model proposes a monolayer sorption with a heterogeneous energetic distribution of active sites, accompanied by interaction between adsorbed molecules. The general form of this model is presented as:

$$q_e=K_fC_e^{1/n}$$

Where ,K_f stands for adsorption capacity, n for adsorption intensity, q_e is the amount of Cr^{+6} adsorbed at equilibrium, mg/l,C_e is the residual concentration of Cr^{+6} in solution, mg/l.

Table (2): *Results for equilibrium parameters for the adsorption of Cr(IV) from waste water onto the prepared solids.*

Solids	C_i, mg/l	Removal., %	q_e, mg/g	C_e, mg/l	log q_e	logC_e	log(1+1/C_e)	1/C_e	C_e/q_e
Ni/ SiRI	10.00	60.43	6.04	3.95	0.78	0.59	0.09	0.25	0.65
	20.00	54.98	10.99	9.00	1.04	0.95	0.04	0.11	0.81
	50.00	30.98	15.49	34.51	1.19	1.53	0.01	0.02	2.22
Ni/SiRII	10.00	75.52	7.55	2.44	0.87	0.38	0.14	0.40	0.32
	20.00	68.00	13.60	6.40	1.13	0.80	0.06	0.15	0.47
	50.00	50.87	25.43	24.56	1.40	1.39	0.01	0.04	0.96
Ni/SiRIII	10.00	77.66	7.76	2.23	0.89	0.34	0.16	0.44	0.28
	20.00	70.56	14.11	5.88	1.14	0.76	0.06	0.17	0.41
	50.00	55.77	27.88	22.11	1.44	1.34	0.01	0.04	0.79
Ni/SiGI	10.00	62.77	6.27	3.72	0.79	0.57	0.10	0.26	0.59
	20.00	60.0	12.00	8.00	1.07	0.90	0.05	0.12	0.66
	50.00	39.98	19.99	30.01	1.30	1.47	0.01	0.03	1.50
Ni/SiGII	10.00	78.52	7.85	2.14	0.89	0.33	0.16	0.46	0.27
	20.00	75.09	15.01	4.98	1.17	0.69	0.07	0.20	0.33
	50.00	57.56	28.78	21.22	1.45	1.32	0.02	0.04	0.73
Ni/SiGIII	10.00	81.60	8.16	1.84	0.91	0.26	0.18	0.54	0.22
	20.00	78.00	15.6	4.40	1.19	0.64	0.08	0.22	0.28
	50.00	60.00	30.00	20.00	1.47	1.30	0.02	0.05	0.66

The values of n and K_f were obtained from the intercept and slope of a plot of log$q_e$versus logC_e from linear form of Freundlich. The Langmuir isotherm model was applied to estimate the adsorption capacity of adsorbents used and suggests that uptake occurs on a homogeneous surface by monolayer sorption without interaction between adsorbed molecules. The linear form of the Langmuir adsorption isotherm is represented as:

$$C_e/ q_e=(1/ b\, Q_o +C_e/Q_o)$$

Q_o, mg /g and b, l /mg are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. The values of $Q_o$and b were calculated from the slope and intercept of the Langmuir plot of C_e versus C_e/q_e.Results of the modeling of the isotherms of Cr^+ sorption by the prepared adsorbents according to Langmuir and Freundlichmodels, are summarized in Table (3). According to the coefficient of correlation obtained, we deducted that the model of Langmuir correlated the experimental data well and better than the Freundlich model for both the adsorbents group. From Table (3) for Ni/SiR and/or Ni/SiG solids presents the estimated parameters of Langmuir and Freundlich isotherm models for the adsorption of the Cr^+ions. According to the Langmuir model, the maximum adsorption occurs when a saturated monolayer of metal ions is reached on both the adsorbents group surface and

the energy of adsorption is constant and no migration of adsorbate molecules on the plane surface of the solids are observed.The constant b in the Langmuir equation relates to the energy or the net enthalpy of the adsorption process . The higher the value of the constant b, the lower the free energy and the process is more favorable because it requires less energy to sustain. The Freundlich isotherm model encompasses on the surface heterogeneity and the exponential distribution of active adsorption sites and their energies . Parameter, n is indicative of intensity of the interaction between the adsorbent –adsorbate, showing how strong the metal ions are attached to adsorbent surface. Table (3)represents the n values between 1.00 and 10.00 for Ni/SiR and/or Ni/SiG adsorbents, which indicate favorable adsorption process and the adsorption intensity was favorable. From the results, it is clear that the value of adsorption efficiency Q_o of Ni/SiR and /or Ni/SiG increased by addition of Ni for adsorption of ions.The observed b value show the endothermic nature of the process involved in the system and values of n are greater than one, indicating also the adsorption is much more favorable.

Table (3) :*Freundlich , Langmuir and D-R models for* the prepared solids.

Solids	Freundlich model			Langmuir model				D-R model			E_a, $kJ.mol^{-1}$
	k_f, $mg.g^{-1}$	n	R^2	Q_0, $Mg.g^{-1}$	b, $l.mg^{-1}$	R^2	R_L	q_D, $mg.g^{-1}$	B_D, $mol^2.k\,J^{-2}$	R^2	
Ni/ SiRI	3.71	2.43	0.83	18.99	0.13	0.99	0.99	17.49	0.41	0.99	1.10
Ni/SiRII	4.26	1.92	0.99	35.29	0.10	0.99	0.99	13.84	0.32	0.95	1.25
Ni/SiRIII	5.12	1.85	0.99	40.04	0.10	0.99	0.99	26.60	0.28	0.88	1.33
Ni/SiGI	3.23	1.85	0.91	27.95	0.08	0.99	0.99	22.66	0.45	0.99	1.05
Ni/SiGII	5.37	1.81	0.95	41.16	0.11	0.99	0.99	31.22	0.31	0.94	1.27
Ni/SiGIII	6.30	1.92	0.95	41.20	0.13	0.99	0.99	32.35	0.27	0.95	1.36

The essential characteristics of Langmuir isotherm can be expressed in terms of dimensionless constant separation factor for equilibrium parameter, R_L which is defined as given below:

$$R_L=1/(1+bC_o)$$

Thus, R_L is a positive number whose magnitude determines the feasibility of the adsorption process. The shape of the isotherms can be predicted by the R_L value. The R_L value between 0 and 1 indicates favorable adsorption of Cr^{+6} onto studied adsorbents. The values of R_L for the studied system at different initial concentrations were found to be in between 0 and 1 which indicate favorable adsorption of Cr^{+6} onto the adsorbents. Langmuir model represents the monolayer adsorption on to a homogeneous surface with a finite number of active sites where as D–R model does not assume homogeneous surface. The D–R adsorption isotherm is represented as:

$$logq_e=log\ q_D-2B_DR^2T^2\ log(\ 1+1/C_e)$$

Where,$q_D$is theoretical saturation capacity, mg.g^{-1}., B_D is a constant related to adsorption energy ,mol^2 kJ^{-2}, R is the gas constant and T is the temperature ,K. The slope of the plot log q_e versus log$(1+1/C_e)$ gives the q_D and B_D values. The values of linear regression coefficient (R^2) are in the range of 0.99 - 0.88, revealing that the experimental data fitted well with the D–R isotherm model. It is revealed that the q_D values are not complying with the previously determined Langmuir isotherm Q_o values. The constant B_D gives an idea about the mean free energy E_D(kJ /mol)of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be calculated from the D–R isotherm constant B_D using following equation:

$$E_D=1/(2B_D)^{0.5}$$

The results so obtained are given in Table (3). The calculated E_D values were found to be 1.20±0.10 kJ /mol for Ni/SiR and/ or Ni/SiG solids. E_D value less than 8.00 kJ/ mol as indicated by our results shows that the adsorption process of Cr^{+6} on the both adsorbent groups follows physical adsorption(Hasany& Chaudhary, 1996). In contrast, if the value of E_D is located in the range of 8.00 − 16.00 kJ/mol, it is the chemical adsorption.

Conclusions

- Ni/SiR and Ni/SiG could be suitable adsorbents for the removal ofCr(VI) from waste waters. The adsorption was found to bestrongly dependent on pH, adsorbent dose, contact time, temperature and initial Cr(VI) concentration.

- Optimum conditions for Cr^{+6} removal were found to be pH =3.00, adsorbent dosage 0.05 g/L of solution and equilibrium time 2.00 h. The experimental data were analyzed using Freundlich, Langmuir and Dubinin–Radushkevich (D–R) isotherm models. It was found that Freundlich and D–R models fitted well.

- The n values between 1.00 and 10.00 for Ni/SiR and/or Ni/SiG adsorbents, which indicate favorable adsorption process and the adsorption intensity was favorable, and the value of adsorption efficiency Q_o of Ni/SiR and /or Ni/SiG increased by addition of Ni for adsorption of ions.The observed b value show the endothermic nature of the process involved in the system and values of n are greater than one, indicating also the adsorption is much more favorable.

- The value of the adsorption energy calculated using D–R isotherm, was 1.20±0.10 kJ /mol for Ni/SiR and Ni/SiG solids and it indicated that the adsorption process was physical in nature. The Ni/SiG solids shows higher capacity for adsorption of Cr(VI) than Ni/SiR solids.

- Application study was also carried out to find the suitability of the process in waste water treatment operation.

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الملخص باللغة العربية

التطبيق البيئي علي النيكل المحمل علي النانو سيليكا المحضرمن المخلفات الزراعية

أميرة سعيد حسن ، عصام محمد عزو، سوزان أحمد حسن ، ماجده عبد الباسط الخرباوي

قسم الكيمياء ـ كلية البنات ـ جامعة عين شمس

تتلخص النقاط الرئيسية التي قام بها البحث على النحو التالي:

● تم تحضير ست عينات من السليكا و المحملة بالنيكل بنسب مختلفة. و قد تم تخضير السليكا النشطة من قشور الارز و ثلاث عينات الاخري من السيلكا جل المحضره من الاثيل اوكسي سليكات بطريقة الصول جل

● كتطبيق بيئي تمت دراسة تقييم كفاءة المواد الصلبة المحضره في إزالة صبغة الكروم سداسي التكافؤ من مخلفات مصنع قها للصناعات الكيميائية و قد لوحظ أن نسبة ازالة الصبغة تعتمد علي درجه حموضة الوسط و التركيز الابتدائي و زمن التلامس ودرجة الحرارة .

● تم حساب طاقة التنشيط لامتزاز الصبغة علي سطح المواد الصلبة المحضره ووجد انها تساوي $1.20\pm0.10 \ kJ \ /mol$ وهذا يدل ان الامتزاز ذات طبيعة فيزيائية .