Int. J. Environ. Sci. Technol. (2015) 12:857–864
DOI 10.1007/s13762-013-0460-4

ORIGINAL PAPER

Application of manganese nodules leaching residue for adsorption of nickel(II) ions from aqueous solution

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Received: 30 January 2013 / Revised: 7 May 2013 / Accepted: 24 November 2013 / Published online: 10 December 2013

Abstract Deep ocean manganese nodules are significant futuristic resource of copper, nickel and cobalt. After recovery of these valuable metals, a huge quantity of residue (~70 % of ore body) is generated. In the present paper, investigations carried out for the application of washed manganese nodule leaching residue (*w*MNR) for the removal of nickel (Ni) ions from aqueous solution by adsorption are described. Several parameters have been varied to study the feasibility of using *w*MNR as potential adsorbent for remediation of Ni(II)-contaminated water. The adsorption kinetics followed pseudo-first-order equation, and the rate of adsorption increased with solution temperature. Kinetics data of Ni(II) adsorption were also discussed using diffusion models of Webber–Morris and Dumwald–Wagner models. The equilibrium data were best fitted into Langmuir adsorption isotherm, and the maximum adsorption capacity was found to be 15.15 mg g^{-1} at pH 5.5 and temperature 303 K, which decreased to 10.64 mg g^{-1} upon raising the solution temperature to 323 K. The activation energy for Ni(II) adsorption onto *w*MNR was 9.56 kJ mol^{-1} indicated physical sorption. Desorption studies showed successful regeneration of adsorbent and recovery of Ni. This process can be utilized for removal and recovery of Ni from the industrial effluent.

Keywords Adsorption · Manganese nodule leached residue · Heavy metals · Nickel · Diffusion

Introduction

Heavy metals are notorious for their hazardous effect on living beings. These metals enter into healthy bodies through contaminated air, water or soil. Though some elements such as iron (Fe), zinc (Zn), copper (Cu), cobalt (Co), chromium (Cr), manganese (Mn) and nickel (Ni) are needed in small quantities for human metabolism, they may be toxic at higher levels (INSA 2011). Among these elements, Ni is a toxic heavy metal used in various applications or industries such as silver refineries, electroplating, zinc base coating, ferro-nickel smelters and storage battery industries (Nicolaidou and Nott 1990; Cempel and Nikel 2006; INSA 2011). According to Agency for Toxic Substances and Disease Registry (ATSDR), the exposure of the surrounding general population to Ni is mainly through oral intake, primarily through water and food, as a contaminant in drinking water or as both a constituent and contaminant of food (ATSDR 2005). The chronic toxicity of Ni to human and the environment has been well documented. For example, high concentration of Ni(II) causes cancer of lungs, nose and bone. Soil contamination by Ni has been reported as threat to the crop productivity of the whole world as Ni is readily transported from roots to overground plant tissue (ATSDR 2005). Several methods have been developed for remediation of Ni-contaminated water bodies such as chemical precipitation, electrocoagulation, ion exchange and adsorption (Zheng et al. 2009; Jadia and Fulekar 2009). Compared with precipitation and ion exchange treatment, adsorption has been viewed as most versatile and effective method for removing heavy metals from aqueous solutions. Adsorbents based on zeolites, activated carbon, etc., have been adequately utilized for adsorptive removal of Ni (Zheng et al. 2009). However, for selection of an adsorbent, cost has been considered a prime

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factor, and hence, use of low-cost adsorbents such as activated carbon produced from wastes, clay, biosorbents, fly ash, etc., has also been studied over the years for removal of Ni from contaminated water (Hussein et al. 1998; Wang et al. 2008; Paul et al. 2012; Moodley et al. 2011).

Metallic oxides (Fe, Mn, Al, etc.), especially of waste category, are of much interest due to their effectiveness toward adsorptive remediation of heavy metals from contaminated aqueous bodies (Lee 1974; Green-Pederson et al. 1997). Residues generated after hydrometallurgical treatment of manganese nodules or polymetallic sea nodules contain oxides/oxyhydroxides of Fe, Mn, aluminum (Al) and silicon (Si) with a reasonable porosity and surface area (Jana et al. 1999a, b; Acharya et al. 1999). The residues generated in [NH_4OH/$(NH_4)SO_4$] leaching have been effectively utilized as an adsorbent for a variety of heavy metals (Parida et al. 2004; Das and Jana 2006). In the present work, studies were aimed to investigate the sorption characteristics of residue, generated in the reduction roasting–ammoniacal leaching of manganese nodules (Jana et al. 1999a), for the removal of Ni(II) ions from its aqueous solution. Several parameters such as adsorbent dose, Ni concentration, pH of solution, temperature and time have been varied to investigate the Ni adsorption characteristics of leached sea nodules residue. The research work descried in this paper was carried out in the Council of Scientific & Industrial Research-National Metallurgical Laboratory (CSIR-NML), Jamshedpur, India, in June–September 2012.

Materials and methods

Adsorbent

The adsorbent material, i.e., manganese nodule leaching residue (MNR), was obtained from large-scale trial of reduction roasting–ammoniacal leaching of manganese nodules at CSIR-NML, Jamshedpur, India. To remove the entrapped leach liquors, MNR was washed with deionized water with 1:10 solid-to-liquid ratio and stirring for 2 h. The washed manganese nodule residue (*w*MNR) was separated by filtration followed by air-drying for several days for subsequent characterization and adsorption studies.

Solution preparation

The synthetic stock solution of Ni(II) of 1,000 mg L^{-1} was prepared by dissolving nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) in deionized water, and the solution was made slightly acidic by adding a few drops of nitric acid (HNO_3) to prevent hydrolysis of the solution. Solutions of 0.01 M HNO_3 and 0.01 M sodium hydroxide (NaOH) were used for pH adjustment with the help of digital pH meter (Toshniwal CL46) after calibration using National Bureau of Standards (NBS) buffers. And 0.1 N potassium nitrate (KNO_3) was used to maintain the ionic strength in all the adsorption experiments. All the chemicals were Merck-AR grade.

Sample characterization

For chemical analyses, a weighed quantity of MNR or *w*MNR was digested in acid (HCl/HNO_3 mixture), dehydrated, redissolved in hydrochloric acid (HCl) (1:1) and filtered. The dehydrated silica was estimated gravimetrically while major and minor constituents in the filtrate were analyzed by conventional wet chemical methods (Vogel 1978) and atomic absorption spectrometer (Perkin Elmer AAnalyst 400), respectively. Surface area measurements were conducted using Quantachrome 4000E surface area analyser (Nova Instruments, USA). Size analysis was carried out in Malvern Mastersizer after ultrasonic liberation of particles.

Adsorption experiments

For kinetic studies, typically, 100 mL of Ni solution at desired concentration with appropriate amounts of adsorbent was taken in a 250-mL stoppered conical flask. The required pH was adjusted, and flask was then mechanically shaken (120 strokes min^{-1}) using a water bath shaker, which was maintained at desired temperatures. Samples were withdrawn at certain time interval, and the solid adsorbent was separated by filtration/centrifugation. The remaining nickel in the filtrate was analyzed by atomic absorption spectrometer (PerkinElmer AAanalyst400). The amount of Ni per gram of *w*MNR, Q_t (mg g^{-1}), was calculated using Eq. (1).

$$Q_t = \frac{(C_o - C_t)V}{w \times 1000} \tag{1}$$

where C_o and C_t are the initial Ni(II) concentration (mg L^{-1}) and Ni(II) in solution, respectively, V is the volume of solution in mL, and w is the mass of sorbent in gram.

The equilibrium adsorption experiments were carried out to investigate the effect of various parameters, such as pH of the adsorbate solution (3–8), initial Ni(II) concentration (5–100 mg L^{-1}), adsorbent dose (0.25–5.0 g L^{-1}) and temperature (303–323 K) under fixed equilibration time obtained by kinetic experiments. For the desorption studies, Ni-loaded *w*MNR was transferred into 250-mL deionized water adjusted to different pH values in the range

Table 1 Chemical analysis of manganese nodule residue (MNR) and washed residue (*w*MNR)

Element/radical	Chemical composition, % by mass	
	MNR	*w*MNR
Mn	25.66	26.11
Fe	9.92	10.19
SiO_2	15.28	16.44
Al_2O_3	3.53	3.54
S	0.37	0.08
P	1.34	0.19
NH_4^+	0.30	Not found
Moisture	8.96	6.18
LOI	18.85	17.01

of 2–9 maintained by 0.1 M HNO_3 and 0.1 M NaOH and shaken for 2 h at 303 K. The sorbent was filtered or centrifuged, and the filtrate was analyzed to determine the concentration of the desorbed metals. Desorption (%) was calculated using Eq. (2).

$$\text{Desorption}\,(\%) = \frac{\text{Amount of metal ion desorbed}}{\text{Amount of metal ions adsorbed}} \times 100 \qquad (2)$$

Results and discussion

Adsorbent characterization

Detailed chemical analysis of manganese nodule residue (MNR) and washed residue (*w*MNR) is given in Table 1. Mn, Fe and Si are the major constituents along with lime (CaO), magnesia (MgO) and alumina (Al_2O_3). After washing, marginal changes in wt% of Mn, Fe, silica (SiO_2), carbon (C), but significant loss of sulfur (S) and phosphorous (P), are observed. Removal of S and P during washing suggested that part of them are present in soluble form generated from roasting of manganese nodules. Thus, washing of MNR was found to be necessary prior to adsorption studies. The pH_{pzc} and specific gravity of *w*MNR were found to be 6.5 and 3.1, respectively. The surface areas of MNR and *w*MNR are found to be 60.9 and 66.7 $m^2\ g^{-1}$, respectively. The marginally higher surface area of *w*MNR is presumably due to an increased number of accessible pores on washing out adsorbed species from MNR. Particle size analyses of *w*MNR revealed very fine granulometry with mean particle diameters (d_{50}) of 17.8 µm.

Effect of pH

The solution pH is an important parameter that affects adsorption of heavy metal ions. The adsorption of Ni(II) was studied over the pH range of ~3–9, and the results are shown in Fig. 1.

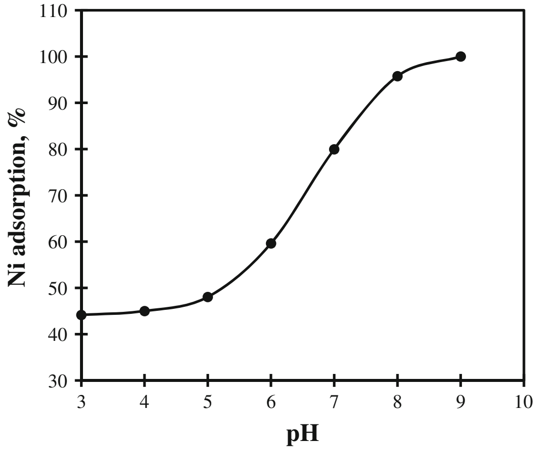

Fig. 1 Effect of pH on Ni(II) adsorption on *w*MNR. Conditions: [Ni(II)] 50 mg L^{-1}; temperature 303 K; time 2 h; *w*MNR 1,000 mg L^{-1}

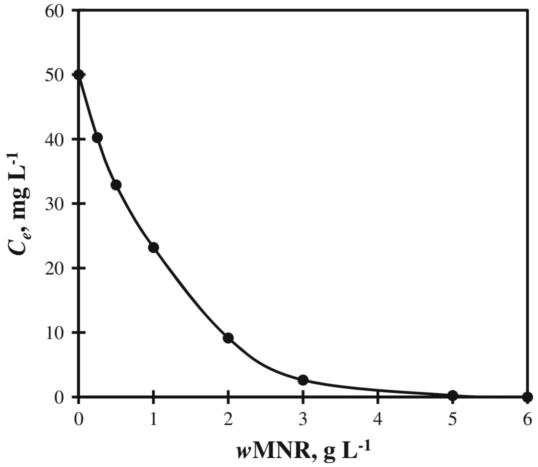

Fig. 2 Effect of weight of *w*MNR on equilibrium concentration (C_e) of Ni(II). Conditions: [Ni(II)] 50 mg L^{-1}; temperature 303 K; time 2 h; pH 5.5

It is seen that the adsorption of Ni(II) increases with increase in pH. Increase in adsorption with pH of solution may be attributed to competitive binding between H_3O^+ ions and Ni(II) ions at the *w*MNR surface. As pH value increases, the competing effect of H_3O^+ ions decreases and the positively charged Ni(II) ions get adhere to free binding sites. The other important factor, which might contribute to the higher adsorption of metal ions with increased pH, is the pH_{pzc} of *w*MNR. When the solution pH exceeded pH_{pzc}, the metal species are more easily attracted by the negatively charged surface of adsorbent, favoring accumulation of cationic metal species on the surface and thus promoting adsorption (Boujelben et al. 2009).

Effect of adsorbent dose

Adsorption of Ni(II) with varying adsorbent dose, carried out to assess the effect of adsorbent on Ni(II) removal, is

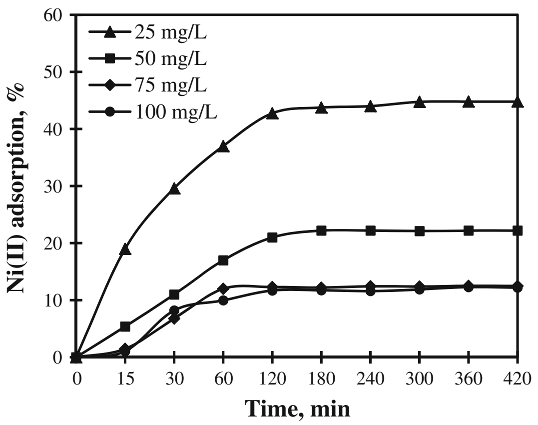

Fig. 3 Effect of time and initial concentration on Ni(II) adsorption onto *w*MNR. Conditions: pH 5.5; temperature 303 K; *w*MNR 1,000 mg L^{-1}

presented in Fig. 2, where equilibrium Ni(II) concentration, i.e., C_e, is plotted against *w*MNR dose. The results show that the equilibrium concentration of Ni(II) decreases with increase in the weight of *w*MNR, which is 40 mg L^{-1} Ni(II) for 0.25 g L^{-1} *w*MNR and negligible for 5.0 g L^{-1} of *w*MNR addition. Increase in *w*MNR dose provides higher surface area and active sites for adsorption of Ni(II) and ultimately responsible for more uptake of Ni(II).

Effect of time and initial concentration on adsorption

The time course of Ni(II) adsorption onto *w*MNR at varying initial concentrations is given in Fig. 3. The Ni(II) adsorption increases with time and attains equilibrium at ~120 min irrespective of initial concentration of Ni(II) ions. Similar equilibrium time has been reported for Ni(II) adsorption onto Turkish fly ash (Bayat 2002); however, numbers of systems are reported to have equilibrium achieved between 15 min and 1 h (Boujelben et al. 2009; Hasar 2003; Suryan and Ahluwalia 2012; Green-Pederson et al. 1997). On the other hand, higher equilibrium adsorption times of about 4 h are reported for chitin (Hema et al. 2011)- and pine tree material (Argun et al. 2010)-based adsorbents. The amount of adsorption is found to decrease with the increasing initial Ni(II) concentration. The Ni(II) removal at equilibrium is 43, 21, 12 and 11 % for 25, 50, 75 and 100 mg L^{-1} initial Ni(II), respectively. For a fixed dose of adsorbent, the decrease in adsorption with increasing Ni(II) concentration is primarily due to the availability of limited number of site for adsorption. However, uptake of Ni(II) onto *w*MNR is seen to be markedly increased.

Adsorption kinetics

The adsorption data obtained from time variation studies were fitted into reaction-based as well as diffusion-based models (given in Table 2) to investigate the kinetics of Ni(II) adsorption by *w*MNR (Lagergren 1898; Ho 2004, 2006; Ho and McKay 1998). The kinetics data were fitted into these models, and interestingly, all the four plots obtained were almost linear (figures not shown). The corresponding rate constants and correlation coefficient values calculated from slope and intercept of the kinetic plots are given in Table 3. On the basis of regression coefficient values, it is concluded that Ni(II) sorption onto *w*MNR can be approximated more appropriately by the pseudo-first-order kinetic model than by the second-order kinetic model. Higher regression coefficient values obtained from Webber–Morris and Dumwald–Wagner models (Table 3) also indicated possible role of diffusion within particles during Ni(II) sorption. In addition, slope in the intraparticle diffusion given by Webber and Morris was found to start from origin and hence may be regarded as the rate-limiting step (Srivastava et al. 2006).

The kinetics of the Ni(II) adsorption was found to be improved by increasing the temperature from 303 to 323 K as evident from rate constants for pseudo-first-order model (Table 3) though equilibrium Ni(II) uptake was adversely affected, presumably an indication of physisorption (Srivastava et al. 2006). Another evidence in support of physical sorption of Ni(II) onto *w*MNR was obtained when activation energy of Ni(II) adsorption was calculated using the following expression:

$$k_1 = k.e^{\left(-\frac{E_a}{RT}\right)} \tag{3}$$

where k_1 is the rate constant for pseudo-first-order kinetics (g mg^{-1} min^{-1}), k the temperature-independent factor (g mg^{-1} min^{-1}), E_a the activation energy of sorption (kJ mol^{-1}), R the universal gas constant (8.314 J mol^{-1} K) and T the solution temperature (K). The activation energy of Ni(II) adsorption onto *w*MNR was calculated from the slope of straight line obtained by plot between ln k_1 and $1/T$. The magnitude of the activation energy has been commonly used as the basis for differentiating between physical and chemical adsorption. The activation energy for Ni(II) adsorption onto *w*MNR was found to be 9.56 kJ mol^{-1} for 25 mg L^{-1} initial concentrations, confirmed that the Ni(II) ions are physically adsorbed onto the *w*MNR surface (Srivastava et al. 2006).

Adsorption isotherms

The equilibrium adsorption data were fitted into the linearized form of isotherm models proposed by Langmuir (Eq. 4) and Freundlich (Eq. 5) models (Das and Jana 2006).

$$\frac{C_e}{q_e} = \frac{1}{bQ^o} + \frac{C_e}{Q^o} \tag{4}$$

$$\ln q_e = (1/n) \ln C_e + \ln K_f \tag{5}$$

Table 2 Kinetic models for adsorption rate calculations

Kinetic models		Plot	Slope	Intercept
1. Reaction based				
Pseudo-first-order	$\ln(q_e - q_t) = \ln q_e - k_1.t$	$\ln (q_e - q_t)$ Vs t	k_1	$\ln q_e$
Pseudo-second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e t}$	t/q_t Vs $1/t$	$\frac{1}{q_e}$	$\frac{1}{k_2 q_e^2}$
2. Diffusion-based				
Webber–Morris model	$q_1 = k_{id}.t^{1/2} + I$	q_t Vs $t^{1/2}$	k_{id}	I
Dumwald–Wagner model	$\log(1 - F^2) = \frac{-kt}{2.303}$	$\log\ (1 - F^2)$ Vs t	k	

Table 3 Kinetic model rate constants for Ni(II) adsorption on *w*MNR at different temperatures

Temperature (K)	Pseudo-first-order model			
	k_1 (min^{-1})	q_e (mg g^{-1})	r^2	
303	0.0284	9.64	0.999	
313	0.0331	7.38	0.999	
323	0.0359	6.29	0.998	
	Pseudo-second-order model			
	k_2 (g mg^{-1} min^{-1})	q_e (mg g^{-1})	V_o (mg g^{-1} min^{-1})	r^2
303	0.004	0.563	12.165	0.998
313	0.005	0.310	7.886	0.995
323	0.005	0.174	5.882	0.986
	Webber–Morris model			
	K_{id} (mg g^{-1} h$^{0.5}$)	Intercept, C	r^2	
303	9.268	0.265	0.993	
313	5.945	−0.004	0.998	
323	4.284	−0.153	0.998	
	Dumwald–Wagner model			
	K	r^2		
303	−0.010	0.993		
313	−0.012	0.993		
323	−0.013	0.989		

where C_e is equilibrium concentration (mg L^{-1}); q_e is amount adsorbed at equilibrium (mg g^{-1}); b is Langmuir isotherm constants related to affinity of adsorbent toward metal ion and Q^o is adsorption maxima or adsorption capacity (mg g^{-1}). The K_f and $1/n$ stand for empirical constants related to adsorption capacity and intensity, respectively. The calculated parameter from Langmuir plot of C_e versus C_e/q_e and Freundlich plot of $\ln q_e$ versus $\ln C_e$ (figure not shown) is given in Table 4.

The Langmuir model is more likely applicable due to higher correlation coefficients, suggesting possible monolayer coverage of Ni(II) on the surface of *w*MNR. Further, the value of Q^o, which is a measure of adsorption capacity, decreased with the rise in temperature. This is also supported by the findings from kinetic studies, where uptake of Ni(II) showed decrease with temperature (Table 3). Dimensionless separation factor, R_L, measure of favorability of adsorption, was calculated using Eq. (6).

Table 4 Langmuir and Freundlich isotherm model parameters and coefficients for adsorption of Ni(II) on *w*MNR

Temp (K)	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg g^{-1})	b (mg L^{-1})$^{-1}$	R^2	K_f (mg g^{-1})	1/n	R^2
303	15.15	0.16	0.994	3.48	0.34	0.914
313	13.33	0.08	0.993	2.06	0.41	0.952
323	10.64	0.09	0.996	1.78	0.35	0.937

$$R_L = \frac{1}{1 + bC_o} \tag{6}$$

where C_o is the initial metal concentration (mg L^{-1}) and b is the Langmuir parameter. The conditions $R_L > 1$: unfavorable; $R_L = 1$: linear; $0 < R_L < 1$: favorable; and $R_L = 0$: irreversible reported in literature (Zheng et al. 2009) when compared with the calculated values of R_L (0.098–0.553) revealed that the adsorption of Ni(II) on *w*MNR was favorable and reversible.

Release of metal ions

Release of metal ions from leached residue during Ni(II) adsorption was also investigated to assess the secondary contamination by the *w*MNR itself at different pH of solution. Figure 4 shows that some amount of metal ions, especially Mn ions, leached out into adsorbate solution. The release of Cu, Co and Fe is, however, not included in the figure, as their release was negligibly small and detectable only at pH < 3. It may also be noted that the release of Mn^{2+} was significantly lowered with increasing solution pH. The other metal ions released were Ca and Mg only, which were decreased with increasing pH and were negligibly small at pH ~5–6. These values are well within

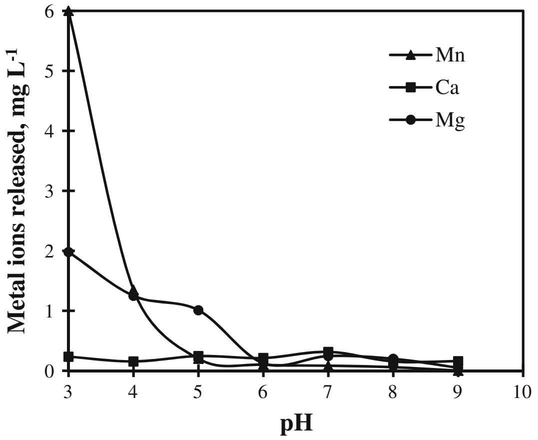

Fig. 4 Release of different metal ions during adsorption of Ni(II) at varying pH. [Ni(II)] 50 mg L^{-1}; temperature 303 K; *w*MNR 1,000 mg L^{-1}; time 2 h

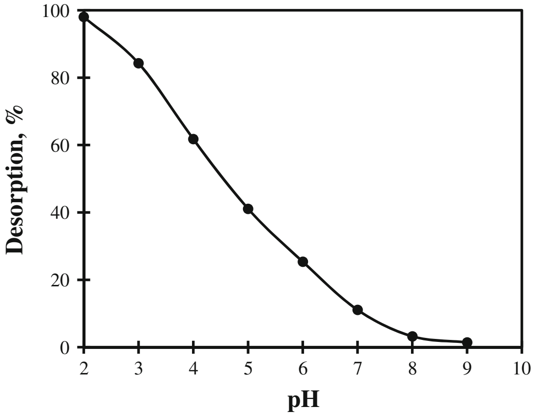

Fig. 5 Desorption of loaded Ni(II) ions from *w*MNR at varying pH

the permissible limit set by World Health Organisation (WHO 2008) for different metal ions in drinking water.

Loading capacity

The Q^o, i.e., maximum loading capacity value, for Ni adsorption on *w*MNR as determined by Langmuir isotherm data was found to be 15.15 mg g^{-1} at 303 K (Table 4). The increase in temperature adversely affected the uptake of Ni(II) onto *w*MNR, decreasing loading capacity to 13.33 mg g^{-1} at 313 K and further down to 10.64 mg g^{-1} at 323 K. This indicated exothermic nature of reactions taken place during adsorption. A comparative account of the adsorption capacities of some adsorbents and *w*MNR for removal of Ni^{2+} are given in Table 5. The *w*MNR has loading capacity almost equal to clinoptilolite (Sprynskyy et al. 2006) but significantly higher than that for most of the adsorbents listed in Table 5 except modified pine bark (Argun et al. 2010) and the activated carbon produced from almond husk (Srivastava et al. 2006). Thus, *w*MNR can also be effectively considered as potential adsorbent for Ni(II) removal from contaminated water.

Elution/adsorbent regeneration studies

The overall metal removal efficiency of an adsorbent also depends on the metal elution/desorption efficiency of the adsorbed metals. Elution/desorption efficiency was defined in this study as the ratio of the mass desorbed from the adsorbent to the mass adsorbed onto the adsorbent. The deionized water employed as eluent and elution of adsorbed Ni(II) ions were investigated in pH range of 3–9. Like the adsorption, desorption of Ni(II) was also seen to strongly depend upon the pH of the solution (eluent) as shown in Fig. 5. As shown, Ni(II) ions were easily eluted from the *w*MNR; elution efficiencies of about 98 % were achieved at pH ≈ 2, and thereafter, the elution of Ni(II) ions progressively decreases with increasing pH of the eluent. Efficient elution indicated that Ni(II) interaction on *w*MNR is completely reversible and the Ni(II) ions are physically adsorbed through electrostatic attraction or weak bond, which could be desorbed to regenerate *w*MNR effectively.

Table 5 Comparative adsorption capacity of some adsorbents for Ni(II) ions in aqueous solution

Adsorbents	pH	Initial metal ion concentration (mg L^{-1})	Adsorption capacity (mg g^{-1})	Reference
Amino phosphate chelating resin	–	–	5.39	Meng (2005)
Natural coated sand	7	30–110	1.08	Boujelben et al. (2009)
Almond husk (activated carbon)	5	25–250	30.769	Hasar (2003)
Paper mill sludge	4.5	5–100	7.861	Suryan and Ahluwalia (2012)
Modified pine bark	8	1–100	20.58	Argun et al. (2010)
Modified pine cone	8	1–100	1.67	Argun et al. (2010)
Clinoptilolite	6.2	10–800	15.55	Sprynskyy et al. (2006)
Fly ash	6	–	2.89	Jadia and Fulekar (2009)
*w*MNR	5.5	5–100	15.15	Present work

Conclusion

The application of *w*MNR, i.e., residue generated from reduction roasting–ammonia leaching of manganese nodule, for the adsorption of Ni(II) from aqueous solution has been examined. The percentage removal of Ni(II) depends on the initial Ni(II) ions concentration and decreases with increase in initial Ni concentration. The adsorption was fairly rapid, and equilibrium was achieved within 2-h contact time. The adsorption capacity increases with the increase in pH of solution. The percentage removal of Ni(II) increases rapidly with increase in the adsorbent dose. The decrease in the equilibrium adsorption of Ni(II) with increasing temperature indicates that the Ni(II) ions removal by adsorption on *w*MNR is exothermic in nature. Langmuir isotherm shows better fit to adsorption data than the Freundlich model indicating monolayer sorption of Ni(II) onto *w*MNR. The monolayer loading capacity of 15.15 mg g^{-1} was obtained at 303 K, which decreased to 13.33 mg g^{-1} at 313 K and down to 10.64 mg g^{-1} at 323 K. The experimental data were fitted by the pseudo-first-order kinetic model. The intraparticle diffusion given by Webber and Morris model was found to be rate-limiting step with slope starting from origin. The adsorbent was successfully regenerated by eluting the adsorbed Ni(II) ions.

Acknowledgments The authors wish to thank the Director, CSIR-NML, Jamshedpur, for his permission to publish this paper.

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