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Potassium bromate modification of the granular activated carbon and its effect on nickel adsorption

D. Satapathy · G. S. Natarajan

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Abstract Granular Activated Carbon (GAC), a commercial adsorbent for the removal of heavy metals was treated chemically with potassium bromate for it's surface modification and it's adsorption capacity was investigated with nickel ions. There was an increase in the adsorption capacity of the modified carbon by 90–95% in comparison to the raw granular activated carbon towards nickel ion adsorption. Potassium Bromate oxidation treatment was employed for a period of about 30 mins initially followed by 60 mins and the oxidized carbons were adsorbed with nickel ions. Metal sorption characteristics of as received and modified activated carbons were measured in batch experiments. Batch adsorption was successfully modeled by Langmuir Isotherm Model which indicates monolayer adsorption. The adsorption isotherms also fit well to the Freundlich Model. Effects of pH of initial solution, time of oxidation and mode of treatment on the adsorption process were studied. Experimental results showed that metal uptake increased with an increase in pH and oxidation time. The samples were characterized by Scanning Electron Microscope (SEM) studies and surface area analyzer.

Keywords Granular Activated Carbon (GAC) · Nickel · Potassium bromate · Surface treatment · Isotherm · Kinetics · Batch experiments

Introduction

The increasing contamination of urban and industrial wastewaters by toxic metal ions is a growing environmental problem. These inorganic micro pollutants are of considerable concern because they are non-biodegradable, highly toxic and have a probable carcinogenic effect. If directly discharged into the sewage system they may seriously damage the operation of biological treatment as well as make the activated sludge unsuitable for application to agricultural land. It is a global problem and necessary precautionary measures should be taken today onwards to protect the environment around us in a safe way.

Heavy metal removal from wastewater usually involves precipitation, ion exchange, membrane process, electrochemical techniques, biological process that require the use of expensive chemicals and may sometimes cause disposal problem. In recent years the adsorption on granular activated carbon has attracted attention because of its effectiveness for the removal of dissolved heavy metal ions at trace quantities. Adsorption on granular activated carbon for the removal of organic and inorganic pollutants has been recognized as the best available technologies (BAT) by the United States Environmental Protection Agencies (USEPA).

G. S. Natarajan: Director, Priyadarshini Institute of Engineering and Technology, Nagpur.

D. Satapathy (✉) · G. S. Natarajan
Regional Research Laboratory, Bhubaneswar-751013,
Orissa, India
e-mail: deba1997@yahoo.com; dsatapathy@rrlbhu.res.in

The adsorption on granular activated carbon has many other advantages : (i) The rate of adsorption and the capacity vary significantly for different potentially hazardous compounds and different commercial carbons. The capacity and the rate are both important determining factors in the design and operational criteria. (ii) The capacity and the rate of adsorption for many organic and inorganic pollutants are affected significantly by the presence of other organic/inorganic substances and by the conditions of the solutions such as temperature and pH.

Nowadays several researchers (Chingombe et al., 2005; Strelko and Malik, 2000; Satapathy et al., 2004; Xiao and Thomas, 2004) are emphasizing on the modification of the carbon surface to make the process cost effective. Modification can be performed by adsorbing foreign organic compounds on the surfaces of the carbons. It is well known that activated carbons are able to effectively adsorb organic compounds in aqueous solutions. This chemical behavior leads to immobilization of organic compounds on the carbon surface (Ravindran et al., 1999) impregnated 8-hydroxyquinoline (oxine) on an activated carbon to improve the adsorption capacity of carbon towards cadmium, lead and zinc (Lakov et al., 1999) modified activated carbons with 3–methyl-1-phenyl-pyrazolore-5 (MPP) and found the copper adsorption was enhanced by 50% (Gardner et al., 1996) modified the surface of the carbon fibers using nitric acid for enhancement of metal adsorption. Oxidation of carbon fibers and subsequently refluxing it with NaOH was carried out by Wu et al. (1995) for increase in the metal adsorption capacity of the carbon fibers.

Nickel is moderately abundant and is the twenty second most abundant element by weight in the earth's crust. It is mostly found along with sulphides of iron or copper (Lee, 1991). Daily intake of nickel from food is 100–300 μg/day in most countries. Critical organs for nickel exposure in humans are their respiratory system, especially the nasal cavities, sinuses and the skin. Exposure to nickel has been known to cause occupational asthma in metal-plating workers (McConnell et al., 1973; Malo et al., 1982). Nickel carbonyl is the most acutely toxic nickel compound. Poisoning can lead to headache, vertigo, nausea, vomiting and severe pneumonia. Chronic irritative effects observed in nickel refinery and nickel plating workers include rhinitis, sinusitis, perforations of the nasal septum and bronchial asthma. High risks have been reported in nickel refinery workers and workers involved in processes with exposure to soluble nickel e.g. nickel sulphate (Magnus et al., 1982), often combined with some exposure to nickel oxide.

The main objective of this paper was to correlate the adsorption performance of Potassium Bromate modified activated carbon with the unmodified activated carbon. Moreover the work was carried out keeping at the aim to develop a cost effective process for nickel adsorption from their aqueous solutions.

Experimental

Apparatus

A Digital Spectrophotometer Type-166 (Systronics India Ltd.) with matched cells of 1 cm optical path length was employed for all absorbance measurements. A mechanical shaker (Remi Model No. RS-24, Remi Instrument Ltd., Mumbai) was used for agitating solutions in experimental bottles provided with a timer. A digital pH Meter (ELICO, Pvt. Ltd., Hyderabad) was used for maintaining the pH of the solution as and when required. All glass wares were 'A' class and were washed thoroughly and rinsed with distilled water and dried in an air oven before use.

Reagents and chemicals

All the reagents and chemicals used for the entire work were of A.R. Grade (E. Merck/S.D. Fine). In the present work Filtrasorb-400 (M/s Calgon Corporation, Pittsburgh, USA) was used. Nickel Sulphate hepta hydrate (E.Merck India Ltd.) was used for the preparation of standard nickel solution and it was diluted proportionately to prepare the experimental solution. Bromine water, ammonia solution and DMG (Dimethylglyoxime) used in the experiment were of Analytical Grade. Potassium Bromate from E. Merck India Ltd. was also used for oxidizing the carbon surface.

General procedure

Standardisation and characterisation of raw and modified Filtrasorb-400

In the present work granular activated carbon namely Filtrasorb-400 was used and was subjected to the following process before its use in the experiment. The carbon was first stirred in boiled distilled water carefully without any attrition followed by cold distilled water. The process was continued till all the fine powdered activated carbon were removed and the supernatant liquid became almost colourless. It was dried in an air oven for about 4 hours at a temperature of 108°C ± 2°C. It was then transferred to a dessicator containing fused calcium chloride where it was allowed to equilibrate for almost a fortnight and was denoted by RAC. The F-400 RAC was also characterised by FT-IR spectra and also subjected to Scanning Electron Microscope (SEM) studies in order to ascertain the type of pores existing. The FT-IR spectra were recorded on a NICOLET Magna-IR550 spectrometer series II while the SEM studies were carried out on a SEM Cambridge Stereoscan S250 MK III.

Modification of Filtrasorb-400 carbon surface with Potassium Bromate

The process of modification of carbon surface with potassium bromate is called chemical modification and involved the following steps. A saturated solution of potassium bromate was prepared by dissolving 5 g of $KBrO_3$ in 100 ml. of double distilled deionised water. The as received granular activated carbon (RAC) were placed in a round bottom flask well equipped with a reflux condenser and a thermometer. The saturated $KBrO_3$ solution prepared earlier was introduced into the flask and was heated in a water bath for about 30 mins. It was then cooled and the carbons were removed from the flask, washed with sufficient distilled water till all the adhering bromates were removed. It was called as modified activated carbon 1 and was designated as MAC1. The modified carbon MAC1 was used for the equilibrium study with the nickel ions. Similarly another set of modified activated carbon was prepared by refluxing the raw carbon with potassium bromate for about one hour with the above experimental arrangement. This was called as modified activated carbon 2 and was denoted as MAC2.

Preparation of the solution of Nickel Ions and it's estimation

A standard stock nickel solution was prepared by taking 0.2808 g of hydrated Nickel Sulphate (E-Merck India Ltd.) and dissolving it in 100 ml of distilled water. 10 ml each of this stock nickel solution was titrated against standard 0.01 M EDTA solution following the standard procedure for the estimation of nickel. Working standard solutions were prepared by appropriate dilution of stock solution. The dilute nickel solution of the concentration range of 10^{-4} M was used for standard Beer's law plot. The amount of nickel in solution was determined colorimetrically using the standard Beer's law plot (Vogel, 1982).

Experimental arrangement for carrying out adsorption equilibrium studies

All adsorption equilibrium experiments were carried out at a fixed temperature of 25°C ± 1°C using a thermostat bath in a batches of four to six units at a time. Each batch consisted of a one liter round bottom Borosil flask and a motor impeller assembly, which enabled vigorous stirring of the solid liquid system by a two bladed Teflon stirrer. Actual experimental runs involving adsorption equilibrium studies consisted of first weighing the required quantity of carbon in a weighing bottle and transferring the same to the experimental flask. 200 ml of the experimental solution was then carefully introduced. The stirrer was then introduced and the whole arrangement stirred at the desired bath temperature (25°C ± 1°C) till the equilibrium was reached. Aliquots of 5 ml of nickel ion solution were then withdrawn from the flask and analysed colorimetrically for nickel ion concentration. Adsorption isotherms were carried out at a $\mathrm{pH} = 5$ of the initial nickel ion solution.

Experimental procedure for studying the kinetics of adsorption of nickel

The procedure for kinetic studies of nickel ion uptake by grades of GAC was carried out utilizing a one liter flask containing GAC of a given weight. 400 ml of the nickel ion solution of known strength was introduced carefully into the experimental flask which was then held in the thermostat bath at 25°C ± 1°C. The solution in the flask was stirred at a RPM of 1000. The time of addition of carbon was noted as time $t = 0$. There after aliquots of 2.5 ml samples of the experimental nickel ion solution from the flask were withdrawn at regular intervals of time by momentarily interrupting the stirrer. The first four readings were taken at 15 min time intervals, the next two readings were taken at 30 min intervals and the final two readings were taken at a time interval of one hour. The concentration of nickel ion at start C_o and C_t at time t in milligrams/liter in the sample was estimated using the standard calibration curve.

Results and discussion

Characterisation of as received F-400 carbon and Potassium Bromate modified carbon

The as received F-400 GAC and the potassium bromate modified F-400 GAC were subjected to Scanning Electron Microscope (SEM) studies to observe if there is any change on the surface of the carbon. The SEM photographs of both the carbon are given in Fig. 1. After potassium bromate treatment has occurred, the surface were pitted and fragmented i.e. the etching effect was more noticeable as can be seen from Fig. 1. Those imperfections were not found on the surface of the untreated carbon. The untreated GAC surface has the longitudinal spinning process related grooves. Whereas the longitudinal spinning process related grooves were slightly removed from the treated GAC and a canal connecting to micropores on the surface might have been provided. These act as a route for entering metal ions into the micropores.

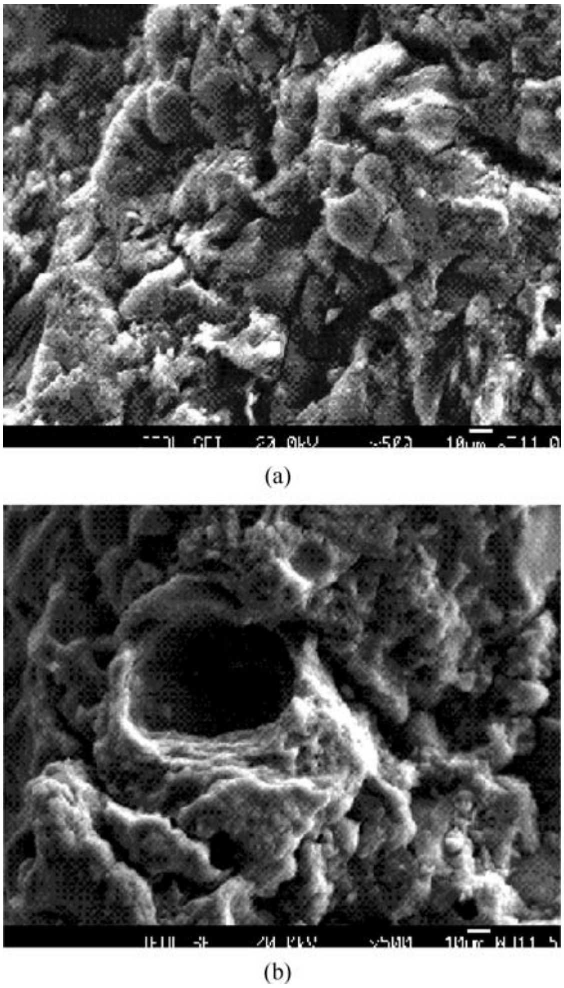

Fig. 1 Scanning Electron Micrographs (SEM) of carbon surface (a) RAC (b) MAC2

It was expected that when raw granular activated carbon was treated with potassium bromate solution, the surface of the carbon might have been oxidized. This was because at the time of heating, the atomic oxygen [O] is evolved which helps in oxidation.

$$KBrO_3 \Leftrightarrow KBr + O_2 + [O]$$

Due to the above oxidation processes more no. of surface groups (—OH, —COOH) are formed which is established by IR spectroscopy. Figure 2 depicts the FTIR spectra of raw and oxidized granular activated carbon. The peaks at 1577 cm^{-1} and 1195 cm^{-1} may be assigned to carbon skeleton. The peaks at 1712 cm^{-1} correspond to the (>C=O) functional groups. The signal at 1385 cm^{-1} may be attributed to O—H bending deformation in carboxylic acid. This shows mostly carboxylic acid groups (—COOH) are formed on the carbon surface rather than hydroxyl (—OH) groups. These functional groups attract more no. of nickel ions thereby increasing the nickel adsorption.

Adsorption isotherm

Equilibrium isotherm equations are used to describe experimental sorption data, with the equation parameters often providing some insight into the sorption mechanism, surface properties as well as the affinity of the sorbent. The Langmuir model is probably the best known and most widely applied sorption isotherm equation. The Langmuir isotherm is valid for monolayer sorption onto a surface containing a finite number of identical sites. The model assumes uniform energies of sorption onto the surface and no transmigration of the sorbate over surface. The Langmuir model has been employed successfully in many real sorption processes and may be expressed as (Bajpai et al., 2000; Agley et al., 2000; Ho and McKay, 1999; Cooner and McQuillan, 1999;

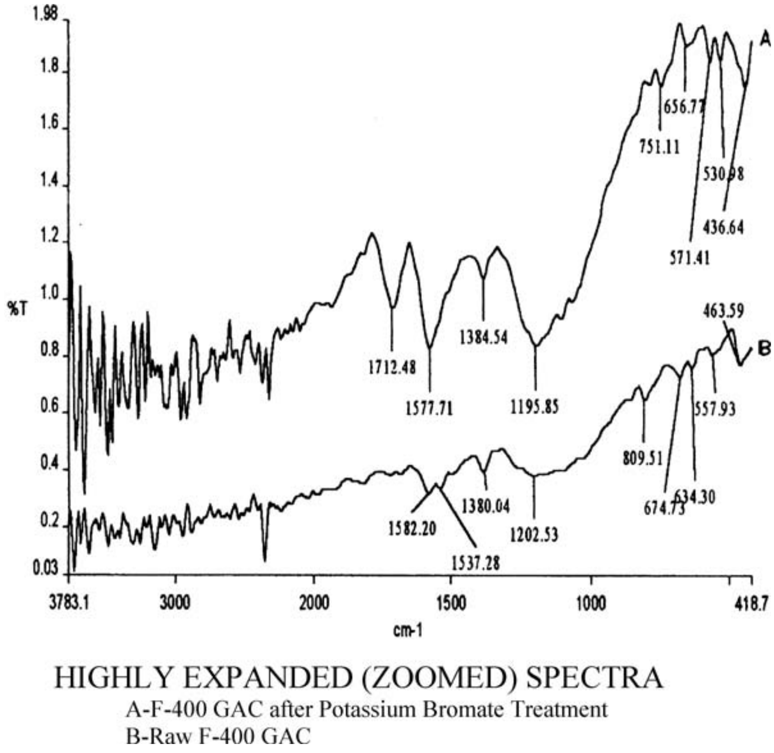

Fig. 2 FTIR spectra of Potassium Bromate modified F-400 GAC and raw F-400 GAC

Sakadevan and Bavor, 1998).

$$\frac{1}{q_e} = \frac{1}{Q^0 \cdot b \cdot C_e} + \frac{1}{Q^0} \tag{1}$$

Where, C_e is the equilibrium concentration of Ni^{+2} ion (mg/l), q_e is the amount of Ni^{+2} ion adsorbed at equilibrium in mg/g, Q^0 is the amount adsorbed corresponding to the formation of a monolayer on the adsorbent and 'b' is the Langmuir constant. From the above Eq. (1), a plot of $1/q_e$ versus $1/C_e$ should be linear if Langmuir operations were operative permitting calculation of Q^0. However it must be cautioned that the Langmuir equation may not always be strictly valid over the range of concentrations used in the present work and only a mathematical analysis of the experimental data would substantiate this point.

Similarly the experimental data were verified with the Freundlich adsorption model over a range of concentrations and can be expressed in the following Eq. (2).

$$\mathrm{Log}\, q_e = \log K + \beta \log C_e \tag{2}$$

Where C_e is the equilibrium concentration of Ni^{+2} ion (mg/l), q_e is the amount of Ni^{+2} ion adsorbed at equilibrium in mg/g, K and β are Freundlich constants. The correlation coefficient (R^2) and both the Langmuir and Freundich constants are given in Table 1.

Figure 3 represents the adsorption isotherms of nickel for raw as well as potassium bromate modified

Table 1 Freundlich and Langmuir Isotherm constants

	Freundlich Isotherm			Langmuir Isotherm		
	R^2	K	β	R^2	Q^0	b
RAC	0.9679	0.2490	0.2393	0.9766	2.8620	1.7661
MAC1	0.9595	0.3963	0.4251	0.9927	6.5274	0.6115
MAC2	0.9784	0.5015	0.3199	0.9983	7.0323	0.6610

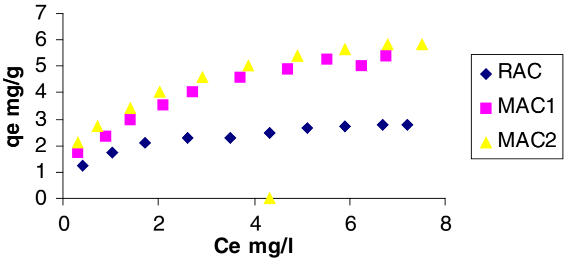

Fig. 3 Adsorption Isotherm for raw and modified granular activated carbons for different concentration of Nickel ions

carbons. A careful observation of these isotherms suggest that there was a considerable increase in the nickel adsorption towards modified carbons in comparision to the raw carbon. It was also seen that when the carbon was oxidized for a longer time period, there was an increase in adsorption for nickel ions. However the difference in the increase in the adsorption of nickel ions for 30 mins and 60 mins oxidation are in the close proximity. Hence one hour oxidation is enough for modifying the carbon surface. The $q_{\max}$ values for RAC, MAC1 and MAC2 are as follows:

RAC $<$ MAC1 $\leq$ MAC2

The results also support the view that the adsorption capacity of the activated carbon was improved by oxidation as potassium bromate act as a very good oxidizing agent.

The Table 1 data shows that the adsorption of Ni^{+2} ions for different concentrations are best fitted to the Langmuir model suggesting monolayer adsorption. The $q_{\max}$ value for MAC2 was found to be 5.81 mg/g compared to the $q_{\max}$ value for RAC which is 2.87 mg/g. It is also seen from the adsorption isotherm that as the oxidation time period increases the amount of nickel adsorption also increases. The $q_{\max}$ value for carbon (MAC1) after 30 mins oxidation with potassium bromate is 5.38 mg/g where as $q_{\max}$ value for carbon (MAC2) after one hour oxidation is 5.81 mg/g.

Kinetic studies

The sorption kinetics may be described by a pseudo-first order equation (Annadurai et al., 2002; Ozarcar and Sengil, 2002; Juang et al., 2001.) The differential form of this equation may be written as :

$$\frac{dq_t}{dt} = K(q_e - q_t) \tag{3}$$

Where q_t (mg/g) is the amount of Ni^{+2} adsorbed at time t, q_e (mg/g) is the amount adsorbed at equilibrium and K is the equilibrium rate constant.

Figure 4 represents the nickel ion adsorption kinetics on raw and modified granular activated carbons. It illustrates that adsorption follows a fast rapid step and a second slow step. Adsorption kinetics for raw granular activated carbon is much slower than that of modified granular activated carbon.

While studying the rate controlling factors in adsorption system, it is worthwhile to study the diffusion process involved in adsorption. Diffusion results from a concentration gradient either in the fluid phase or on the solid phase. when diffusion involves separation of solute from the fluid phase to sorbent phase it is called particle phase diffusion or homogenous diffusion described by an effective diffusion coefficient D. The driving force for pore diffusion which usually occurs within the pores of the adsorbent is due to the concentration gradient of the adsorbate in the pore space, and is characterised by a pore diffusion coefficient D_p. Alternatively surface diffusion which occurs after adsorption due to migration of the adsobed molecules along the pore walls may be represented in terms of gradient of the amount of adsorbate on the pore walls. This is characterised by a surface diffusion coefficient D_s.

In order to explain both the above phenomena and to make a distribution between the surface and pore diffusion (Vermuelen, 1953) proposed the following

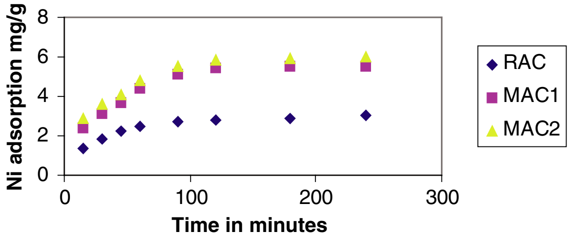

Fig. 4 Adsorption kinetics for raw and modified granular activated carbons for different Nickel ions concentration

Quadratic Driving Force (QDF) expression:

$$\frac{dq}{dt} = \frac{\pi^2 D}{a^2} \times \frac{q^{*2} - q^2}{2q}$$

Where q is the bulk average concentration in the spherical particle, q^* is the surface concentration 'a' is the radius of the spherical particle and 'D' is the diffusion coefficient in the solid phase. In the present investigation the QDF equation has been used to obtain the values of D, the diffusion coefficient and are given below.

Sample identification	D values (cm^2/sec)
RAC	4.338×10^{-8}
MAC1	4.512×10^{-8}
MAC2	4.805×10^{-8}

From this Table it is seen that all the D values are in the range of that encountered for surface diffusion phenomena. Particle phase diffusion could be totally ruled out since then the values would have been much higher.

Mechanism of nickel adsorption

Activated carbon is used by several industries and water treatment plants for the removal of organic and inorganic pollutants from wastewater but the mechanism of sorption of these compounds on activated carbon are still not clear. Some researcher have predicted that organic compounds adsorb through a $\pi - \pi$ dispersion interaction mechanism (Hayder et al., 2003). This is the interaction between π electron clouds of the benzene ring of organic compounds with the π electron clouds of the basal plane of carbon. The mechanism of adsorption of inorganic species on activated carbon is also uncertain. Some people have suggested ion exchange and complex formation as the mechanism of adsorption for inorganic species (Saha et al., 2001; Strelko and Malik, 2000). In our case the proposed mechanism is of similar type. When the carbon is oxidized with potassium bromate , more number of —OH and —COOH groups are formed on the carbon surface which is evidenced from FTIR spectra. Ni^{+2} ions are bonded to these groups to form complexes and gets adsorbed on the carbon surface. Hence more the surface functional groups are formed, more nickel ions will be adsorbed.

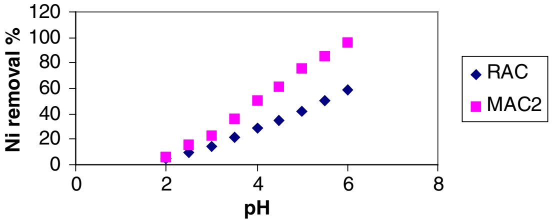

Fig. 5 Effect of initial solution pH on Nickel Adsorption onto both raw and Potassium Bromate modified carbon

Effect of pH

The metal adsorption onto the granular activated carbon is greatly dependent on the pH of the initial metal ion solution. The percentage removal of nickel for both raw as well as potassium bromate modified carbon at different initial pH are depicted in Fig. 5. It was observed that percentage removal of nickel increase with increase in the initial nickel ion solution pH for both raw and oxidized activated carbon. At pH = 2 almost no nickel ions were taken where as at pH = 6 about 95% of the nickel ions were taken by the oxidized carbon and about 55–60% of the nickel ions were taken by the raw activated carbon.

The increase in the removal of nickel ions was apparently due to the increase in the adsorption of nickel ions onto activated carbons, but not due to the nickel ion precipitation because nickel ions in aqueous solutions starts precipitating at pH > 7.2. The nickel adsorption is due to the following reactions:

$$SOH + Ni^{+2} \longrightarrow SO - Ni^{+} + H^{+}$$

At lower solution pH (i.e., when the concentration of hydrogen ion is higher), the above reaction will shift towards left causing less formation of $SO - Ni^{+}$ species. Consequently nickels adsorption was diminished at

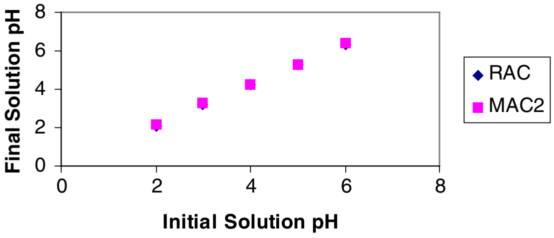

Fig. 6 Change in the initial solution pH and final solution pH for both raw and modified activated carbon

lower pH at around 2. On the otherhand, the effect of hydrogen ions became less important at higher pH (i.e., at lower concentrations of hydrogen ions). As pH was increased, the metal ion removal efficiency was increased.

It was also observed that the final solution pH was increased significantly after adsorption equilibrium was obtained. It is illustrated in Fig. 6. As the activated carbon used (Filtrasorb-400) is an H-type carbon, it can adsorb the hydrogen ions, causing an increase in the solution pH (Chen and Wu, 2000).

Conclusion

The Potassium Bromate modified activated carbon showed a significant improvement in the adsorption of nickel ions compared to that of raw granular activated carbon. This was due to the formation of more no. of surface functional groups. The time of oxidation also plays an important role in surface modification. Increase of oxidation time produced more no. of functional groups on the carbon surface thereby increased the nickel adsorption. Uptake of nickel ions by the raw and oxidized granular activated carbon was enhanced with an increase in solution pH. For raw activated carbon as the pH increased from 2 to 6, the nickel ion removal increased from 5–50% where as for the oxidized carbon as the pH increased from 2 to 6, the nickel removal increased from 5 to 95%. These modified carbons thus can be useful in treating wastewater in a wider pH range. Kinetic studies also indicated that the adsorption took place rapidly in the first hour followed by a period of slower adsorption. The present investigation thus throws light in providing a cost effectiveness of the process for the removal of metal ions through use of modified activated carbons.

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