Adsorption (2008) 14: 711–718
DOI 10.1007/s10450-008-9122-0

Textural and surface chemistry of activated bagasse and its role in the removal of chromium ions from solution

M. Valix · W.H. Cheung · K. Zhang

Received: 30 April 2007 / Revised: 20 February 2008 / Accepted: 18 April 2008 / Published online: 9 May 2008

Abstract The role of the surface chemical and physical properties of activated carbon in the removal of chromium was investigated. This was conducted by fitting the chromium removal by adsorption and reduction to Cr(III) to the physical properties including total surface and pore size of the carbon and its chemical property globally measured using carbon pH. The role of heteroatoms—sulfur, nitrogen, hydrogen and oxygen, to chromium removal was also investigated. This study showed that the structural and chemical properties displayed dual and conflicting properties in removing chromium. As such efficiencies gained in controlling the structure of the carbon are minimal. Optimal carbon properties which exhibited high chromium adsorption included high surface area, large pore size, high quantities of sulfur and nitrogen and minimal hydrogen and oxygen contents.

Keywords Chromium · Activated carbon · Heteroatoms · Textural properties

1 Introduction

Chromium is usually found in industrial wastewaters with the most common sources coming from electroplating, leather tanning and textile industries. In wastewater, chromium can exist both as Cr(III) and Cr(VI). Cr(VI) is considered toxic and prolong exposure can have detrimental effect to human health. In potable waters, Cr(VI) appears as the most stable species due to the aerobic conditions in this environment. Adsorption is considered as an efficient method of removing heavy metals in effluent. Activated carbon has been widely used as the adsorbent. Although carbon adsorption is used globally, this process remains expensive due to the high cost of activated carbon. Recently, production of activated carbon from various agricultural and industrial wastes is of interested. Several studies on chromium adsorption have been conducted using activated carbon obtained from these low cost materials (Babu and Gupta 2008; Baral et al. 2008; Dobrowolski and Stefaniak 2000; Kobya 2004; Kobya et al. 2004; Mise and Shantha 1993; Pereira et al. 2006; Romero et al. 2004; Sharma and Bhattacharyya 2004; Sharma and Forster 1995; Vennilamani et al. 2005; Viraraghavan and Rao 1991).

Removal of chromium metal from solution by adsorption onto activated carbon can be influenced by its oxidation state. Efficient Cr(VI) adsorption requires that chromium be maintained as Cr(VI). However, in the presence of activated carbon, Cr(VI) can be reduced to Cr(III). Although the trivalent form is not considered toxic, Cr(III) adsorption onto activated carbon is met with difficulty because of its high coordination with water (Baes and Mesmer 1976; Okuda et al. 1975). The long term stability of Cr(III) in waste water is of concern. Furthermore, Cr(III) is known to be oxidized by Mn oxides chemically and microbially by reducing bacteria. As such it is important to promote the complete removal of chromium from solution and to limit the reduction of hexavalent chromium to Cr(III).

Significant studies have been conducted in the study of chromium speciation, the role of pH, carbon loading and carbon particle size and their effect on chromium removal but little has been done to investigate the specific characteristics of the carbon which promotes the adsorption of Cr(VI) (Corapcioglu and Huang 1987; Huang and Bowers 1978;

M. Valix (✉) · W.H. Cheung · K. Zhang
School of Chemical and Biomolecular Engineering,
The University of Sydney, Sydney, NSW 2006, Australia
e-mail: mvalix@usyd.edu.au

Perez-Candela et al. 1995). In particular, the separation of the roles of physical and chemical properties of the carbon has not been investigated. This has largely been associated with the difficulty in directly correlating chromium adsorption to physical and chemical properties which varies simultaneously during activation. The separation of the individual effects of each carbon property on the adsorption behavior cannot be precisely conducted.

At present there appears to be no basis for the design or in choosing suitable activated carbon adsorbent specifically for chromium removal. The practice of choosing activated carbon is based on extensive testing of various activated carbons. This study investigates and separates the roles of the physical and chemical properties of activated carbon prepared from bagasse in the removal of chromium. Activated carbons prepared by chemical and physical techniques from bagasse were used as adsorbents in the removal of chromium from waste water. Bagasse is the fibrous by-product derived from the milling of sugar cane.

This study aims to separate and identify the effects of chemical and physical properties of activated carbons that promotes the removal of Cr(VI) by adsorption. The specific interest is in the role of carbon pH and of the heteroatom, sulfur, nitrogen, hydrogen and oxygen in determining the surface acidity of the carbon and the corresponding chromium removal. It is also of interest to establish the enhancement in chromium removal achieved with certain structural characteristics; the total surface area and pore size of the activated carbon. Decoupling these effects is further complicated by the mechanisms involved in chromium removal. The removal of Cr(VI) from waste water by adsorption onto carbon surfaces can be accompanied by the reduction of Cr(VI) to Cr(III) and possible further precipitation to $Cr(OH)_3$ under a neutral or slightly alkaline condition. The precipitation to $Cr(OH)_3$, should this occur within the carbon, would cause severe problems in regenerating the carbon because of the low solubility of this precipitate. The removal of Cr(VI) by activated carbon is a complex process which requires a thorough understanding of the speciation of chromium in solution under various conditions and the role of the carbon properties on the removal of these species.

2 Experimental

2.1 Preparation of the activated carbons

2.1.1 Physical activation

Activation of bagasse in this study involved a two step process: 1) carbonization of bagasse through the use of a dehydrating agent, sulfuric acid followed by 2) gasification with carbon dioxide at 900 °C to develop the extended surface area and porous structure of chars. In the carbonization step, concentrated sulfuric acid was added to bagasse in a 3:4 ratio (by weight). The blend was packed into a Pyrex reactor and heated to 160 °C for two hours with air. Air was metered into the reactor at the rate of 2 liters per min. The carbon was cooled and washed with water until acid free and dried at 110 °C. Details of the gasification unit and procedures are described in detail elsewhere (Otake and Jenkins 1993). Gasification of the carbonized chars was conducted using various concentrations of carbon dioxide including 10, 50 and 100% (v/v).

2.1.2 Chemical activation

The process of chemical activation involves two steps, the first involves the chemical impregnation of the activating agent into the sugar cane fiber, followed by thermal degradation. The chemical activating agents, which included $ZnCl_2$, $MgCl_2$ and $CaCl_2$ were dissolved in water and then added to the sugar cane fiber (bagasse) in a 10:7.5 fiber to activating agent ration by weight. The mixture is well blended and dried at 110 °C. The chemically impregnated fiber was then heated in a furnace between 400 to 500 °C under nitrogen for 2 hours.

2.2 Physical and chemical characterization of the activated carbons

The moisture, ash, pH of the activated carbons was determined according to ASTM tests (ASTM 2007) D2867-95, D2866-94, D3838-80, respectively. The total surface area was determined by N_2 adsorption at 77 K in Quantachrome Autosorb 1-CLP. The carbon, hydrogen, nitrogen and sulfur analyses of activated bagasse were conducted using Elementar vario EL III CHNOS elemental analyzer.

2.3 Chromium adsorption

A 5 mg/L synthetic chromium solution was prepared by dissolving potassium dichromate in distilled water and adjusting the pH to 2. In 500 ml of the chromium solution 0.05 grams of activated carbon was added and kept at room temperature. The pH of the solution was adjusted further with 0.1 M HCl or NaOH to pH 2.0 after the addition of the carbon into the Cr(VI) solution. Samples were taken at various intervals of time, filtered and analyzed. Total chromium concentrations (Cr(VI) and Cr(III)) were determined on a SpectrAA-10 atomic absorption spectrophotometer. Hexavalent chromium concentrations were determined by colorimetric analysis using 1,5-diphenylcarbazide according to the standard methods (American Public Health Association 1980). The absorbance was read on a UV/VIS spectrometer (UNICAM 8625) at a wavelength of 540 nm. Trivalent chromium was measured by subtracting the hexavalent chromium concentration from the total measured using the atomic absorption spectrophotometer.

3 Results and discussions

Adsorption of heavy metals has been found to be influenced by acidity or carbon pH. Basic ($\mathrm{pH} > 7$) and acidic carbon ($\mathrm{pH} < 7$) are generally recognized as having positive and negative surface charges respectively. Basic surfaces promote the sorption of negatively charged metal hydrates or complexes and acidic surfaces promote the sorption of cations. The speciation of metals should be matched to the surface acidity of the carbon to optimize the adsorption of metals. Choosing or designing adsorbents which would efficiently remove chromium requires an understanding of the physical and chemical properties of activated carbon which would promote this removal. Direct correlation of chromium removal to the total surface area (TSA), carbon pH (pH) and pore size (W), however is made difficult by the simultaneous variation of these parameters with activation. To decouple the contribution from each of the carbon characteristics, chromium removal by adsorption and reduction were correlated to the total surface area, carbon pH and pore size using the following equations:

$$\mathrm{Cr(VI)}_{\mathrm{adsorbed}} = k_{\mathrm{Cr(VI)}}[\mathrm{TSA}]^{a}[\mathrm{pH}]^{b}[W]^{c} \tag{1}$$

$$\mathrm{Cr(III)}_{\mathrm{reduced}} = k_{\mathrm{Cr(III)}}[\mathrm{TSA}]^{\alpha}[\mathrm{pH}]^{\beta}[W]^{\delta} \tag{2}$$

where

$\mathrm{Cr(VI)}_{\mathrm{adsorbed}}$ = chromium adsorbed per unit weight of activated carbon on an ash free basis. (mg Cr(VI)/g carbon)
$\mathrm{Cr(III)}_{\mathrm{reduced}}$ = chromium reduced to Cr(III) per unit weight of activated carbon on an ash free basis. This is the resulting Cr(III) measured after adsorption has occurred. (mg Cr(III)/g carbon)
$k_{\mathrm{Cr(VI)}}$ = constant according to model 1
$k_{\mathrm{Cr(III)}}$ = constant according to model 2
TSA = BET N_2 total surface area (m^2/g)
pH = carbon pH
W = median pore size (Å)

The median pore width of the micropores (pore size < 2.0 nm) were estimated by applying the Horvath-Kawazoe (HK) method (Horvath and Kawazoe 1983) which assumes slit pore shapes on the nitrogen adsorption isotherms. Mesopore sized pores (2.0 nm < pore size < 50 nm) were estimated using the Kelvin equation (Rouquerol et al. 1999). The parameters of fitted models 1 and 2 are shown in Table 1.

Table 1 Parameters of models 1 and 2

Cr(VI) adsorption (model 1)		Cr(VI) reduction to Cr(III) (model 2)	
Parameters	Values	Parameters	Values
$k_{\mathrm{Cr(VI)}}$	1×10^{-5}	$k_{\mathrm{Cr(III)}}$	8.5×10^{-7}
a	1.4	α	1.9
b	2.1	β	0.88
c	0.2	δ	0.46

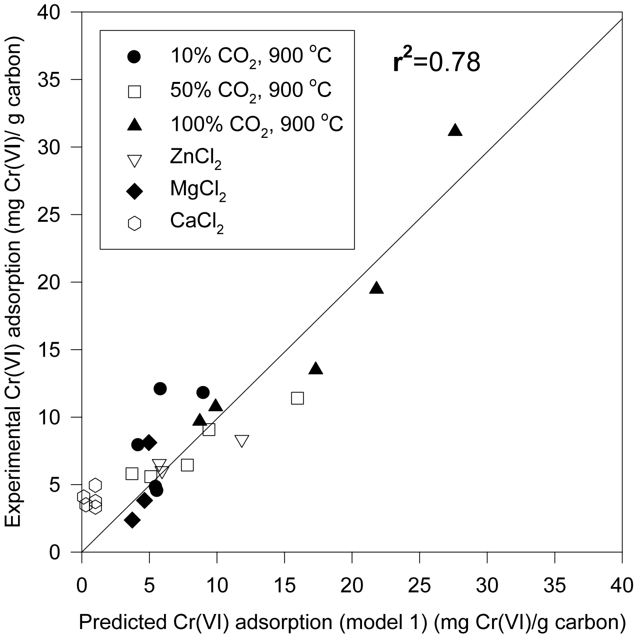

Fig. 1 Regression analysis of fitted chromium adsorption (model 1) and experimental data

Regression analyses of the fitted models 1 and 2 and the experimental data are given in Figs. 1 and 2. The contribution of heteroatoms including sulfur, nitrogen, oxygen and hydrogen to the surface acidity of activated carbon has been widely implied in literature (Bansal et al. 1988; Barton et al. 1997; Benaddi et al. 2000; Corapcioglu and Huang 1987; Leon et al. 1992; Lopez-Ramon et al. 1999; Otake and Jenkins 1993; Sidgwick et al. 1966; Toles et al. 1999). It has been shown that heteroatom sites could be effectively correlated to the experimentally determined carbon pH, if there is no soluble ash (Valix et al. 2006). These previous results suggest that the surface acidity and as such the metal adsorption property of activated carbon could be correlated to its chemical analysis. To investigate further the role of heteroatoms in the removal of chromium, the carbon pH in models 1 and 2 were replaced by concentrations of heteroatoms (wt%) of the activated carbon used in the study. The fitted models (models 3 and 4) are based on similar physical properties of the carbon total surface area, and

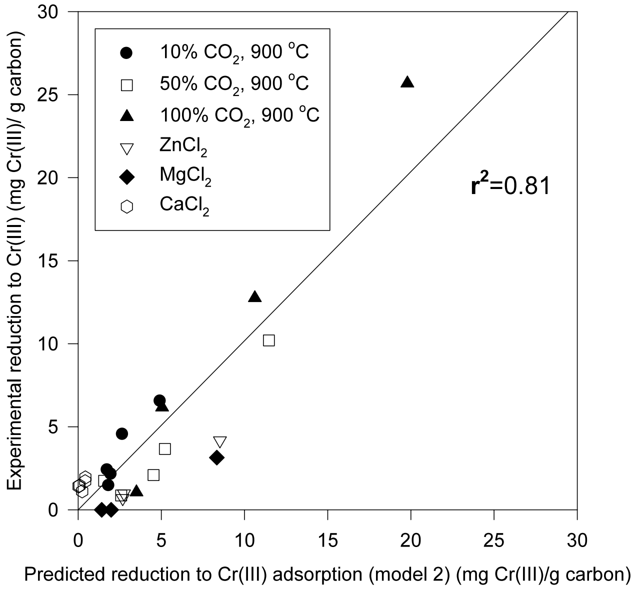

Fig. 2 Regression analysis of fitted chromium reduction (model 2) and experimental data

pore size as used in models 1 and 2. The corresponding models are:

$$\mathrm{Cr(VI)}_{\mathrm{adsorption}} = k'_{\mathrm{Cr(VI)}}[\mathrm{TSA}]^{\eta}[W]^{\mu}[\mathrm{S}]^{\varpi}[\mathrm{N}]^{\sigma}[\mathrm{H}]^{\phi}[\mathrm{O}]^{\iota} \quad (3)$$

$$\mathrm{Cr(III)}_{\mathrm{reduction}} = k'_{\mathrm{Cr(III)}}[\mathrm{TSA}]^{\gamma}[W]^{\lambda}[\mathrm{S}]^{\rho}[\mathrm{N}]^{\upsilon}[\mathrm{H}]^{\zeta}[\mathrm{O}]^{\psi} \quad (4)$$

where

$k'_{\mathrm{Cr(VI)}}$ = constant according to model 3

$k'_{\mathrm{Cr(III)}}$ = constant according to model 4

[S] = sulfur content of the carbon (wt%)

[N] = nitrogen content of the carbon (wt%)

[H] = hydrogen content of the carbon (wt%)

[O] = oxygen content of the carbon (wt%)

The parameters of models 3 and 4 are reported in Table 2. Regression analysis of the fitted models 3 and 4 and experimental data are given in Figs. 3 and 4. As shown, models 1 and 2 gave a regression correlation of r^2 of 0.78 and 0.81 suggesting relatively good fit. However models 3 and 4 have correlations of 0.91 and 0.96. The higher correlations obtained in fitted models 3 and 4 suggest these models provide a better description of the experimental data. These results further confirm that heteroatom concentrations provide an adequate indication of surface acidity and thus chromium adsorption in comparison to conventional carbon pH tests. Experimental carbon pH tests are easily subjected to residual chemicals and extent of washing.

Table 2 Parameters of models 3 and 4

Cr(VI) adsorption (model 3)		Cr(VI) reduction to Cr(III) (model 4)	
Parameters	Values	Parameters	Values
$k'_{\mathrm{Cr(VI)}}$	0.7	$k'_{\mathrm{Cr(III)}}$	5.9×10^{-3}
η	0.46	γ	1.1
μ	0.25	λ	0.73
ϖ	3.7×10^{-2}	ρ	0.12
σ	0.42	υ	0.92
ϕ	−0.53	ζ	−0.38
ι	-2.4×10^{-2}	ψ	-6.2×10^{-2}

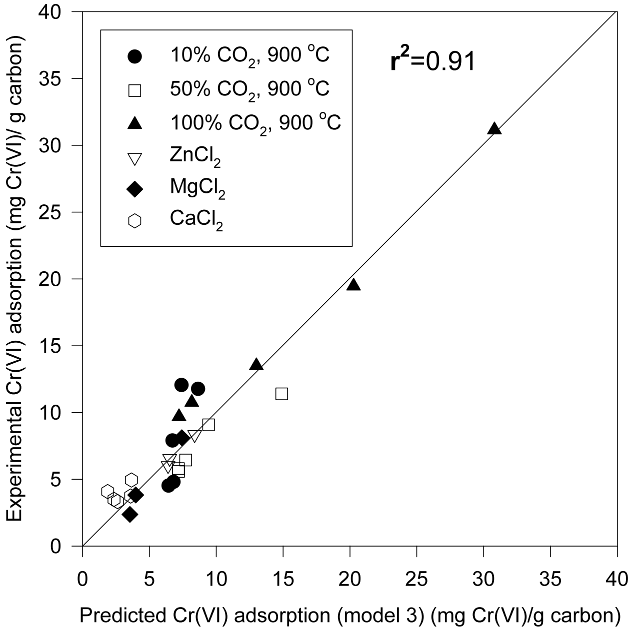

Fig. 3 Regression analysis of fitted chromium adsorption (model 3) and experimental data

The role of the physical and chemical properties of the activated bagasse in the removal of chromium was analyzed according to models 3 and 4. The effect of the structural properties of activated carbon; total surface area and pore sizes, to chromium adsorption by adsorption and reduction is shown in Fig. 5. The effect of the structural properties on the ratio of chromium removal by adsorption and reduction is shown in Fig. 6. As shown increasing total surface area and pore size both promote the adsorption of Cr(VI). However these same properties also enhance the reduction of chromium to Cr(III). This is evident in the decline of the Cr(VI) to Cr(III) ratios in Fig. 6 with increasing surface area and pore size. The increase in adsorption with total surface area would be as expected. Adsorption of the metals is a surface effect which is enhanced both with higher surface area and also higher accessibility provided by the larger pores. The greater reduction of chromium observed

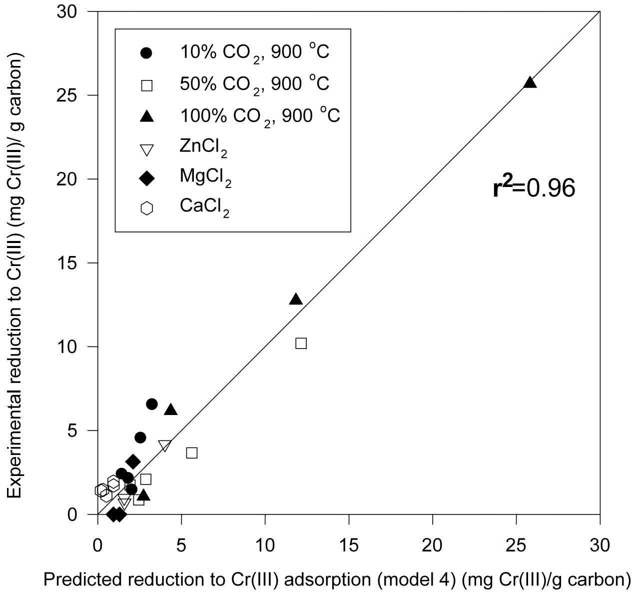

Fig. 4 Regression analysis of fitted chromium reduction (model 4) and experimental data

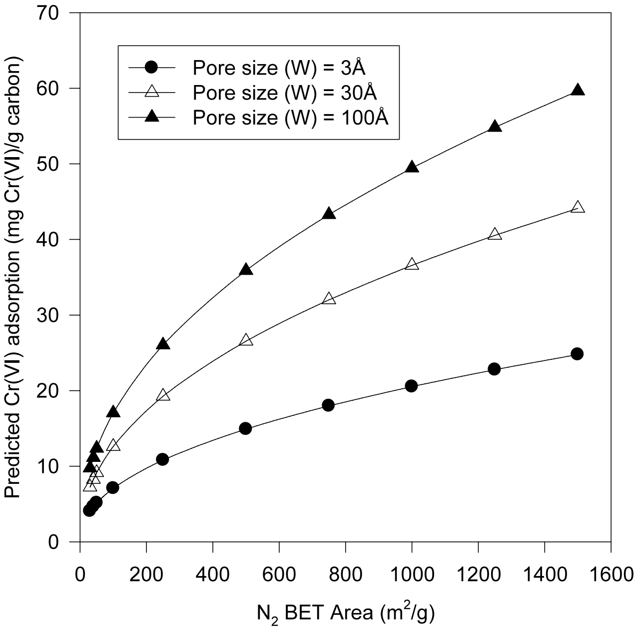

Fig. 5 Predicted Cr(VI) adsorption as a function of carbon total surface area at various pore sizes. Constant parameters: [S] = 0.03 wt%, [N] = 1.3 wt%, [H] = 1.1 wt%, and [O] = 2.8 wt%

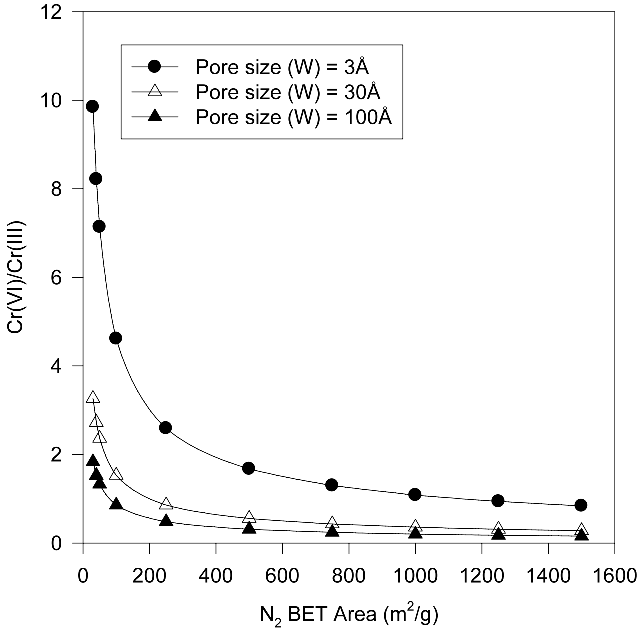

Fig. 6 Relative ratio of chromium removal (Cr(VI)/Cr(III)) as a function of total surface area at various pore sizes. Constant parameters: [S] = 0.03 wt%, [N] = 1.3 wt%, [H] = 1.1 wt%, and [O] = 2.8 wt%

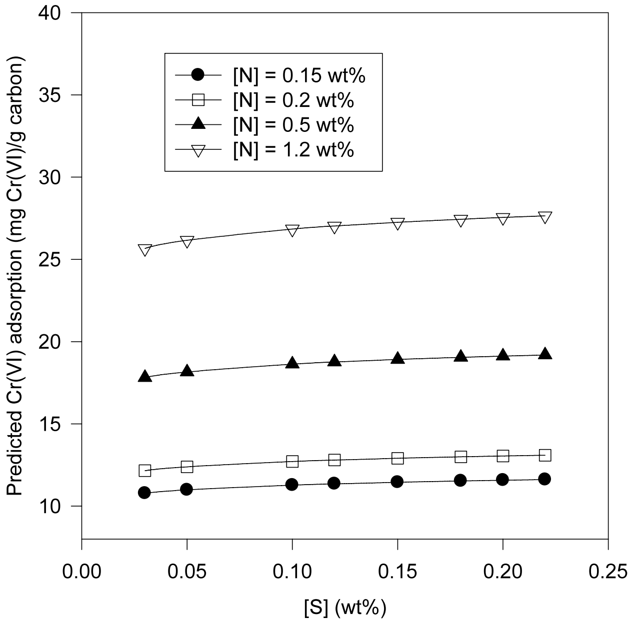

Fig. 7 Predicted Cr(VI) adsorption as a function of sulfur and nitrogen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [H] = 1.1 wt%, [O] = 2.8 wt%

with larger pores is associated with the reductive property of activated carbon. It would be anticipated that the depletion of oxygen and thus the reductive property of carbon would be greater in the inner pores where the surface area is higher and where oxygen diffusion could be limited by restricted flow of the solution and air. The greater accessibility provided by the larger pores allow dichromate ions to diffuse easily into the pores allowing enhanced reduction of these ions to occur.

The effect of heteroatoms in the removal of chromium is analyzed in Figs. 7, 8, 9, 10, 11 and 12. Previous analysis of the contribution of heteroatom to the surface acidity of carbon suggested sulfur and nitrogen sites have basic

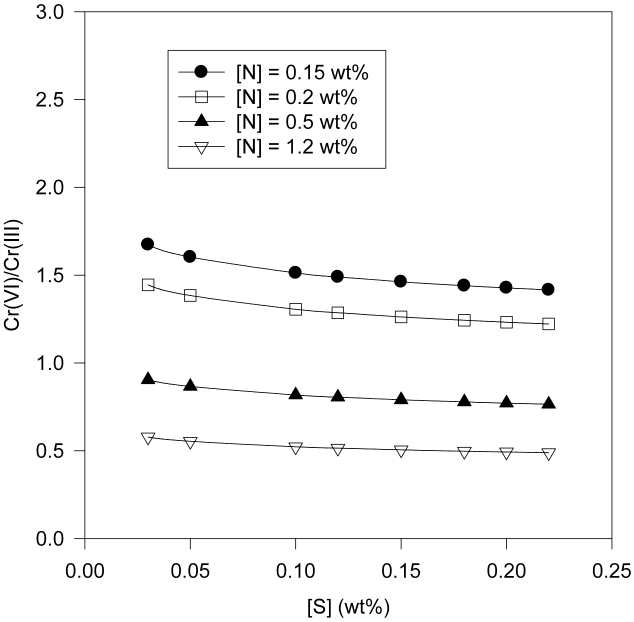

Fig. 8 Relative ratio of chromium removal (Cr(VI)/Cr(III)) as a function of sulfur and nitrogen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [H] = 1.1 wt%, [O] = 2.8 wt%

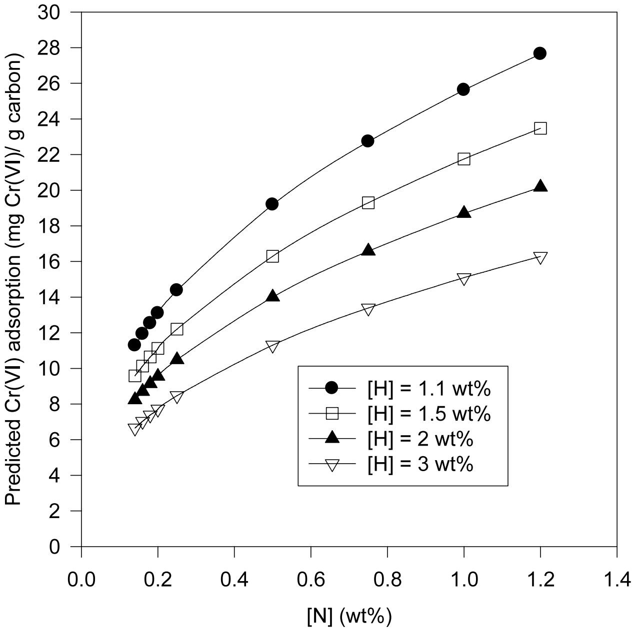

Fig. 9 Predicted Cr(VI) adsorption as a function of nitrogen and hydrogen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [S] = 0.22 wt%, [O] = 2.8 wt%

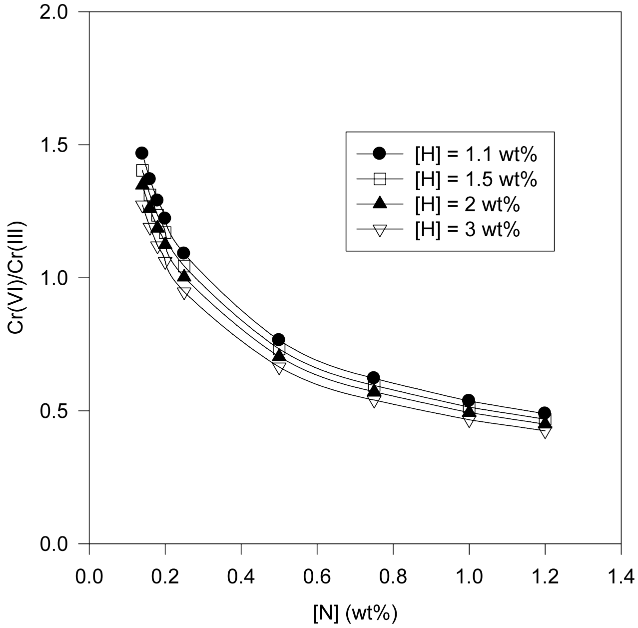

Fig. 10 Relative ratio of chromium removal (Cr(VI)/Cr(III)) as a function of nitrogen and hydrogen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [S] = 0.22 wt%, [O] = 2.8 wt%

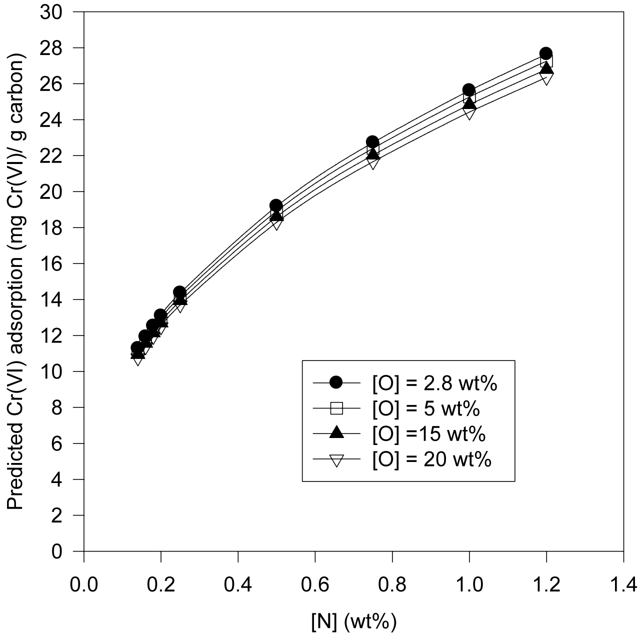

Fig. 11 Predicted Cr(VI) adsorption as a function of nitrogen and oxygen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [S] = 0.22 wt%, [H] = 1.1 wt%

sites providing a net positive charge in the carbon. Hydrogen and oxygen have acidic properties providing net negative charges. These properties are consistent with the observed Cr(VI) adsorption affected by the heteroatom sites. As shown in Fig. 7, increasing the sulfur and nitrogen sites increased the adsorption of negatively charged dichromate ions. Similarly the acidic site attributed to oxygen and hydrogen suppressed Cr(VI) adsorption (see Figs. 9 and 11). Sulfur which promotes adsorption also enhanced chromium reduction (see Fig. 8) and oxygen and hydrogen sites suppressed chromium reduction (see Figs. 10 and 12). In both

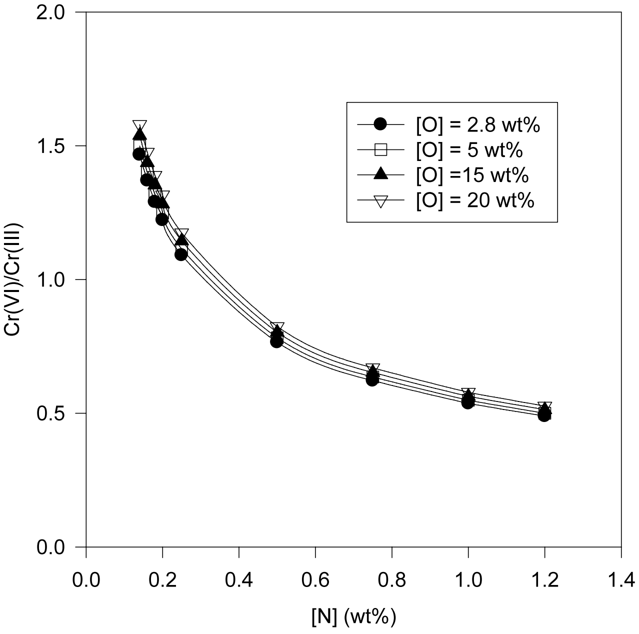

Fig. 12 Relative ratio of chromium removal (Cr(VI)/Cr(III)) as a function of nitrogen and oxygen contents of the carbon. Constant parameters: TSA = 500 m^2/g, $W = 30$ Å, [S] = 0.22 wt%, [H] = 1.1 wt%

reduction and adsorption, the effect of oxygen sites appears less in comparison to hydrogen sites. These results suggest the adsorption and oxidative behavior of heteroatom. Their roles appear conflicting in removing chromium and as such efficiencies gained for example by adding nitrogen sites which promote adsorption can be reduced by its reduction of chromium. Overall it appears chromium removal is optimized with carbon containing high nitrogen and sulfur and minimal oxygen and hydrogen sites.

4 Conclusion

1. Heteroatom sites provide an adequate indication of surface acidity and thus chromium adsorption of activated carbon.
2. Activated carbon exhibiting high surface area and large pore size promotes Cr(VI) adsorption and reduction. Although efficiencies of adsorption are reduced by this conflicting effect in chromium removal, the overall chromium removal is optimal for carbon with large surface areas and pore sizes.
3. Sulfur and nitrogen which have basic properties promote the adsorption of Cr(VI) but also its reduction. Hydrogen and oxygen sites which have acidic property suppress both the adsorption and reduction effects. The conflicting effects of each heteroatom in chromium removal reduce the efficiency of the adsorbents. The effect of the heteroatom in the surface acidity of the carbon is sufficiently significant compared to their oxidative/reductive behavior. This suggests that optimizing the adsorption of chromium will require carbon with basic surface. As such carbon containing high nitrogen and sulfur content and minimal hydrogen and oxygen contents, will satisfy this requirement.
4. Models 3 and 4 are the best-fit models to predict the adsorption and reduction of chromium(VI) on to the activated carbon from bagasse. These models involved the total surface area and median pore size of the produced carbon and the concentration of the heteroatoms on the carbon surface, namely, sulfur, nitrogen, hydrogen and oxygen.

Acknowledgements The authors would like to acknowledge the support of the Sugar Research and Development Corporation, Australia.

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