Environ Sci Pollut Res (2011) 18:75–81
DOI 10.1007/s11356-010-0358-3

RESEARCH ARTICLE

Interaction of Cr(VI) species with thermally treated brick clay

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Received: 12 February 2010 / Accepted: 7 June 2010 / Published online: 19 June 2010

Abstract

Purpose The purpose this research is to investigate the interaction of Cr(VI) species, present as $Cr_2O_7^{2-}$, at ambient temperature with brick clay pre-fired at different temperatures.

Methods A multi-technique approach was used for this investigation. Experiments such as surface titrations, Langmuir and Freundlich adsorption isotherms, mass-firing temperature investigation, scanning electron microscopy, Fourier transform infrared spectra, X-ray fluorescence spectra, and X-ray diffraction were conducted in this investigation.

Results Fired brick clay, which bears a negative charge according to surface titration measurements, shows affinity towards Cr(VI) species despite the negative charge of the source of Cr(VI). The Cr(VI)—brick clay heterogeneous system, which shows the strongest interaction with brick clay fired at 200°C, obeys both the Langmuir and the Freundlich adsorption isotherms with high regression coefficients. Investigation on surface charge, constituents of brick clay, acid treatment of clay particles, and the effect of firing temperature suggests that the reduction of Cr(VI) to Cr(III) by reducing agents present in brick clay makes a significant contribution for adsorption of chromium species followed by subsequent removal. Scanning electron microscopic images support the adsorption of chromium species, and further, many metal ions are released as a result of Cr (VI)—brick clay interaction according to X-ray fluorescence studies.

Conclusion It is concluded that fired brick clay shows strong adsorption capacity on Cr(VI), having the maximum interaction with brick clay fired at 200°C. It is proposed that this methodology be extended for treatment of effluents containing Cr(VI) species.

Keywords Chromium · Brick clay · Adsorption · Surface charge · Interaction

1 Introduction

Undoubtedly, ever-increasing industrial activities have caused heavy metal pollution in the environment, which has already become a global problem. Effective methods, based on the introduction of synthetic chemicals, are available as reliable means of removing toxic heavy metals from the environment (Gyliene and Visiniakova 2008; Jolly et al. 2009). Electrochemical methods have also been tested for treatment of industrial effluents (Babu et al. 2007; Elasayed and Saba 2009). Removal of toxic substances by the use of chemicals could be made limited if natural, environmentally friendly, and low-cost substances are introduced. Among them, naturally occurring clay minerals and saw-dust having strong ion exchange properties have been attempted by many researchers as viable alternatives to chemical adsorbents for the removal of inorganic pollutants (Tertre et al. 2006; Erdem et al. 2008; Eisazadeh 2008; Bajpai and Rohit 2009). Excellent capacity of adsorption, surface specificity and porosity are among many desirable properties of clay minerals toward the removal of chemical pollutants from the environment (Arfaoui et al. 2008).

Removal of pollutants by a solid is associated with strong adsorbent—adsorbate attractions. Adsorption iso-

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Responsible editor: Philippe Garrigues

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therm and spectroscopic investigation on these attractions would be valuable in order to develop suitable models which in turn provide mechanistic information (Sajidu et al. 2008; Ei-mofty et al. 2008; Seneveratne and Priyantha 2009). Designing a suitable model based on experimental observations of a single adsorbate on a pure adsorbent may not be a difficult task. However, the formulation of a generalized model for the interaction of a set of adsorbates with natural matrices containing different clay minerals would definitely be challenging, partly due to many modes of mass transfer from solution phase to solid phase such as adsorption, absorption, ion exchange, and transfer through pin holes and cavities (Juang et al. 2003). Nevertheless, models, even if semi-empirical in nature, designed based on a limited number of experimental observations may be extended for real application, in particular in treatment of industrial effluents by clay-based substances.

There are many reports available on the adsorption of Cr (VI) species by clay minerals and soil types. Among them, adsorption patterns of Cr(VI) species on pure minerals, such as kaolinite, montmorillonite, alumina, and silica, indicate that the mineral surfaces become saturated at a certain concentration (Bois et al. 2003; Fritzen et al. 2006; Bhattacharyya and Gupta 2006). Interaction of Cr(VI) with clay/soil in the presence of organic substances (Deng et al. 2003; Yang et al. 2008), and development of models for adsorption of Cr(VI) by clays modified with heat treatment, acid treatment, and application of pressure, have been attempted (Arfaoui et al. 2008; Juang et al. 2003; Kumar et al. 2008; Weng et al. 2008). Kinetic studies of clay types for adsorption of Cr(VI) (Weng et al. 2008; Venkatraman et al. 2009) and other pre-treatment methods to increase the Cr(VI) removal ability have also been recently reported (Kumar et al. 2007).

Although infrared spectroscopic investigation provides information on bonding type during adsorption/removal of organic species by clay minerals (Kubicki et al. 1997; Seunghun and Xing 2007), practical difficulty in handling the low-wave number end of the finger print region is probably a key factor for lack of infrared data on metal ion removal. Furthermore, difficulty in obtaining well defined X-ray diffraction (XRD) peaks due to the inhomogeneous nature of natural clay types, which contain different types of clay minerals, limits mechanistic investigation despite its necessity.

Although it has been conclusively demonstrated that fired brick clay is suitable for metal ion removal, mechanistic approach for Cr(VI) removal has not been reported to the best of our knowledge. The objective of this study is mainly to investigate surface and equilibrium properties of the Cr(VI)—fired brick clay system, in an attempt to obtain information on the mechanism of Cr (VI)—burnt brick particle interaction. This investigation is aided by the changes in the extent of Cr(VI)—brick clay interaction with the ionic strength and the pH of the medium.

2 Materials and methods

Materials Standard solutions of Cr(VI) (2 to 1,000 ppm) were prepared using analytical grade $K_2Cr_2O_7$ (BDH Chemicals). Unfired brick clay and brick clay fired in the laboratory at 200°C were used for all experiments. It has already determined, through XRD studies, that brick used for this investigation consists of aluminosilicates in additions to normal constituents such quartz and topaz (Priyantha et al. 2009). Different elements present in brick clay (Na, Mg, Al and Fe as major elements, and K, Ca, Ti, Cr, Mn, Cu and Zn as minor elements) have also been determined by X-ray fluorescence and atomic absorption spectroscopic (AAS) measurements. Brick clay samples were separated into appropriate sizes using a set of sieves. All solution experiments were conducted with brick particles of diameter (d) < 1.0 mm at ambient temperature. Fourier transform infrared (FTIR) and scanning electron microscopic (SEM) experiments were conducted with powdered brick clay particles of d < 0.075 mm.

Instrumentation The Carbolite CTF 12/100/900 furnace was used to fire brick clay samples. Spectro-Electronic M Series atomic absorption spectrophotometer was used to measure metal ion concentrations. Centrifugation was done before atomic adsorption measurements to remove suspended particles when turbidity problems were encountered. Surface titration experiments were conducted with Autotitrator (Orion Model 960, USA). FTIR spectra were recorded on Thermo Nicolet Model-Avater 320 FTIR spectrophotometer, while SEM images of brick clay samples were obtained from the Scanning Electron Microscope, JSM-6320F.

Research design Brick, being a natural substance having variable composition, reflects many difficulties in quantitative analysis. Consequently, the method of sampling and the number of trials selected should be given sufficient attention. Representative samples were thus prepared by grinding many pieces of brick clay samples randomly obtained from a bulk, followed by firing at each predetermined temperature. Firing was conducted using the method reported elsewhere (Priyantha and Bandaranayaka 2010).

For surface titrations, a brick clay suspension of fine powder of 5 gL^{-1} in a 0.1 M $NaNO_3$ solution was stirred for 12 h in a closed container to form a homogeneous suspension. The initial pH of the suspension was lowered to a known pH (~4.0) using HNO_3. The resulting solution was then titrated with a standard NaOH solution until the pH reached 9.5. A back titration was carried out using the same HNO_3 solution to ensure the same changes in pH during the titration. The entire procedure was repeated for two more ionic strengths (0.01, 0.001 M).

The amount of Cr(VI) adsorbed/removed by brick clay was determined by treatment of standard Cr(VI) solutions of concentration varying from 2.00 to 100.0 ppm with clay samples fired at 200°C. For these determinations, mixtures of fired brick clay and aqueous Cr(VI) solutions in 1:10 (*w*/*v*) ratio were stirred for 10 min and allowed to settle for 2 h, the supernatant of each mixture was quickly separated, filtered, and atomic absorption measurements were recorded in triplicate. Three brick clay samples were used for each set of experimental conditions, and hence a measurement used for calculations was an average of nine measurements. These measurements were subsequently used for adsorption isotherm analysis. Acid treatment experiments were done by shaking brick clay fired at 200°C with HCl solutions (1:10 *w*/*v* ratio) of different concentrations varying from 0.01 to 12.00 M.

3 Results and discussion

3.1 Surface characterization

Sorption of Cr(VI) by fired brick clay is strongly dependent on the firing temperature showing the maximum sorption for brick clay fired at 200°C, as reported elsewhere (Priyantha and Bandaranayaka 2010). Therefore, many experiments were conducted with brick clay fired at this temperature. Although the extent of Cr(VI) removal by brick clay has many contributions, such as adsorption, absorption, and ion exchange, adsorption of Cr(VI) or its chemically transformed states is probably the initial step of the removal of Cr(VI). As this process is controlled by the surface chemistry of brick clay particles, surface characterisation of brick clay is a necessity in order to investigate the interaction between Cr(VI) and brick clay particles.

Surface titration curves obtained for brick clay fired at 200°C in $NaNO_3$ solutions of different ionic strengths are shown in Fig. 1. The surface charge density for each addition of NaOH, which is needed for the construction of the curves in the figure, was calculated using a standard equation (Dove and Craven 2005). The curves obtained at three ionic strengths do not intersect at a common point, and the curve obtained in 0.1 M $NaNO_3$ is almost parallel to that in 0.001 M $NaNO_3$, indicating that the pH independent surface charge of brick clay particles is more predominant compared to the pH dependant charge. This is further supported by pH independence of the extent of Cr(VI) removal when the medium pH is changed up to 11.0, as reported elsewhere (Priyantha and Bandaranayaka 2010). The point of zero charge can however be approximated to pH 6.5 based on the point of intersection of the two curves obtained in $NaNO_3$ solutions of 0.01 to 0.001 M.

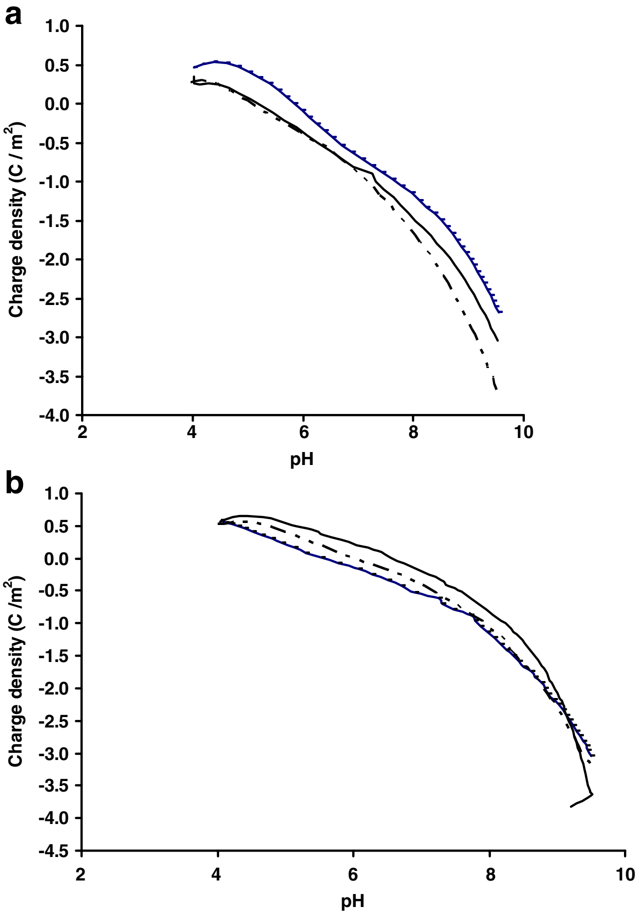

Fig. 1 Surface titration curves of brick clay suspensions, plotted as variation of surface charge density with pH controlled by different additions of NaOH for brick clay fired at 200°C (*top*) and unfired brick clay (*bottom*). Ionic strengths are 0.1 M (*dashed line*), 0.01 M (*solid-dashed line*) and 0.001 M (*solid line*)

The negative surface charge of brick clay particles at neutral pH indicates that direct Coulombic attraction of Cr(VI), which is available as $Cr_2O_7^{2-}$, with brick particles is not possible in contrast to the situation with Cr(III) (Juang et al. 2003). Detailed investigation of the Cr(VI) removal process is thus necessary to understand the underlying mechanism. Although the surface charge curves obtained with raw brick (unfired) clay indicate that the charge on the surface is less negative, adsorption sites are not exposed as no heat treatment is involved. Lower removal of chromium species with unfired clay is thus expected. This is further supported by the mass-firing temperature curve where the initial mass reduction occurs at 100°C due to the vaporization of moisture, exposing sites for adsorption (Fig. 2).

SEM images of powdered brick clay samples fired at 200°C show the presence of different particle sizes, most of which are greater than 100 nm (Fig. 3a). The SEM image of Cr(VI)-treated brick clay samples appears to be different, and some surface particles are even bigger than 200 nm, This observation clearly indicates that brick surface is covered with Cr(VI) species or its transformed states (Fig. 3b). Adsorption of chromium species on brick surfaces is thus confirmed.

3.2 Sorption of Cr(VI) on fired brick clay

The variation of the mass of Cr(VI) removed from solution per unit mass of fired brick clay (*x*) with the initial concentration of Cr(VI) species indicates that the adsorption process on clay fired at 200°C can be identified as type II isotherm (Fig. 4) according to IUPAC classification (Nigel Works Ltd. 2010). This suggests that the adsorption on brick particles show strong adsorbate-adsorbent interactions. Furthermore, the Langmuir and the Freundlich adsorption isotherms within the concentration range from 2 to 100 ppm lead to linear relationships with high regression coefficients of 0.999 and 0.997, respectively (Fig. 5). The Langmuir isotherm describes quantitatively the build up of a monolayer of chemical species on an adsorbent surface which has a limited number of identical adsorption sites. It is given mathematically as,

$$1/x = 1 + (1/K)1/C \qquad (1)$$

where *K* is the adsorption coefficient and *C* is the equilibrium concentration (LeVan and Vermeulen 1981). The magnitude of *K* for the adsorption process under investigation is determined to be 4.47. This is higher than that for pure and modified bentonite clays which show *K* values ranging from 0.3 to 4.0 (Arfaoui et al. 2008). The highest *K* value of 4.0 among many clay types, reported

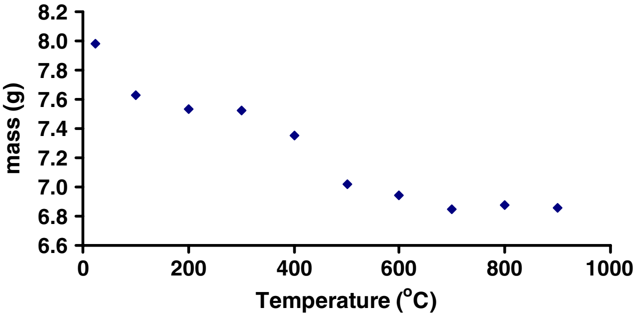

Fig. 2 Variation of mass (g) of brick clay (initial mass 8.0 g, d< 0.075 mm) with firing temperature

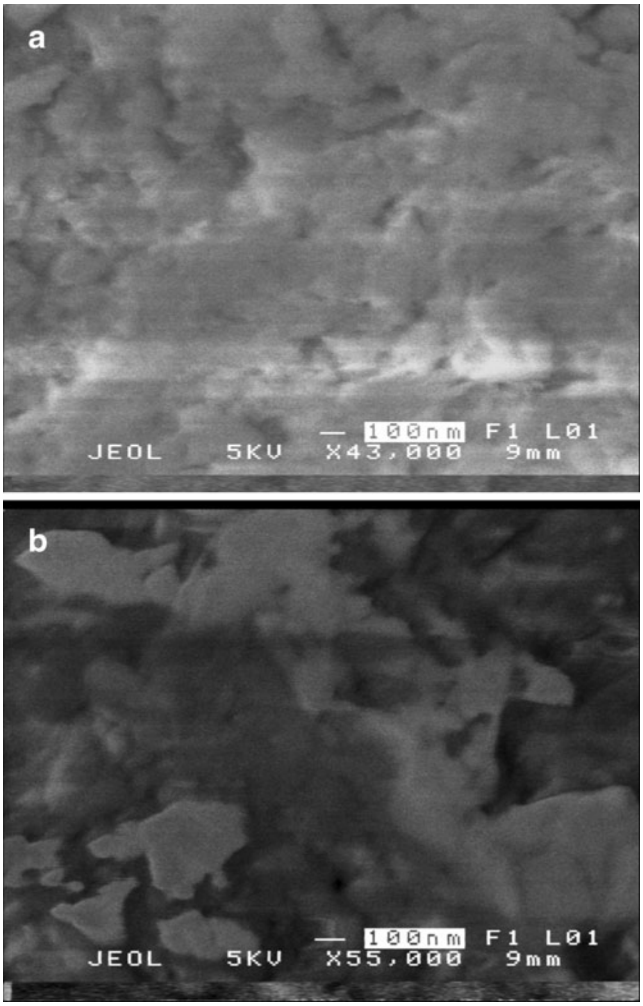

Fig. 3 **a** Scanning electron microscopic (SEM) image of brick clay fired at 200°C and **b** SEM image of the same brick after treatment with 100 ppm Cr(VI) solution (10 min stirring, 2 h settling) followed by rinsing with water and drying

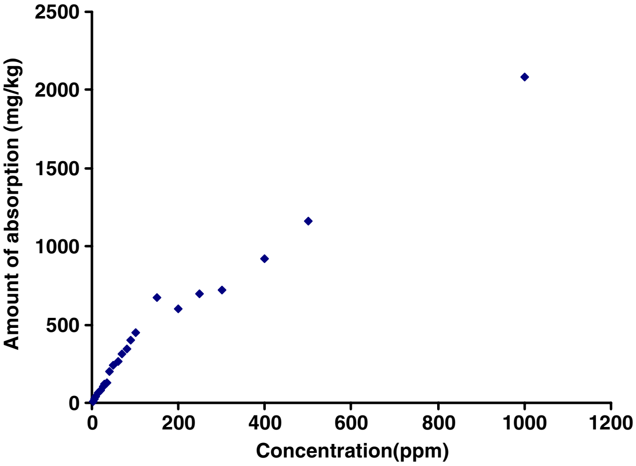

Fig. 4 Variation of the mass of adsorption of Cr(VI) with the initial concentration [5.0 g brick clay, d<1.0 mm, 50.0 cm^3 20 ppm Cr(VI) solution, 10 min stirring, 2 h settling]

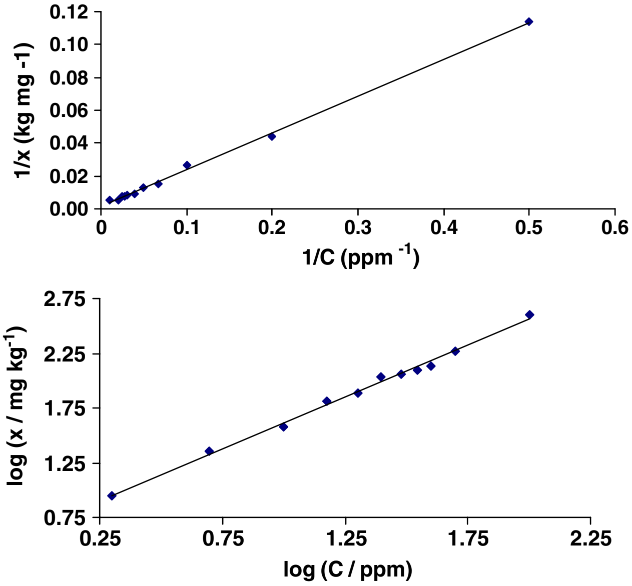

Fig. 5 Lamgmuir isotherm model (*top*) and Freundlich isotherm model (*bottom*) [5.0 g brick clay, d<1.0 mm, 50.0 cm^3 20 ppm Cr(VI) solution, 10 min stirring, 2 h settling]

for purified clay, is the closest to the value determined in this investigation. High values of *K* are indicative of strong adsorption and having the similar magnitudes of the *K* value for purified clay and brick clay fired at 200°C can be argued that the firing process provides enhanced exposure of adsorption sites, equivalent to a purification process.

The Freundlich isotherm, given by,

$$x = K_f C^n \qquad (2)$$

where K_f and n are empirical constants, supports the presence of a large number of adsorption sites associated with different energies where most favorable sites are first filled (LeVan and Vermeulen 1981). Therefore, this model is often employed in real situations such as chromium adsorption on brick particles. The values of the constants n and K_f, determined from the Freundlich plot in Fig. 5, are 0.95 and 4.58, respectively. The n value between 0.0 and 1.0 indicates favorable adsorption (Arfaoui et al. 2008), while the relatively low K_f value suggests that less number of active sites is available. This is further supported by the fact that the Langmuir model shows a better fit as compared to the Freundlich model according to the regression coefficients of the respective linearized plots. It is suggested that adsorbed chromium species in the monolayer undergo ion exchange and other bulk processes with concomitant adsorption of another monolayer from solution. This process of adsorption—ion exchange would empirically be viewed as a multilayer process showing the validity of the Freundlich isotherm. These two isotherms are the most popular two parameter, single component empirical representations of experimental data on adsorption. The validity of both isotherms in this investigation suggests the

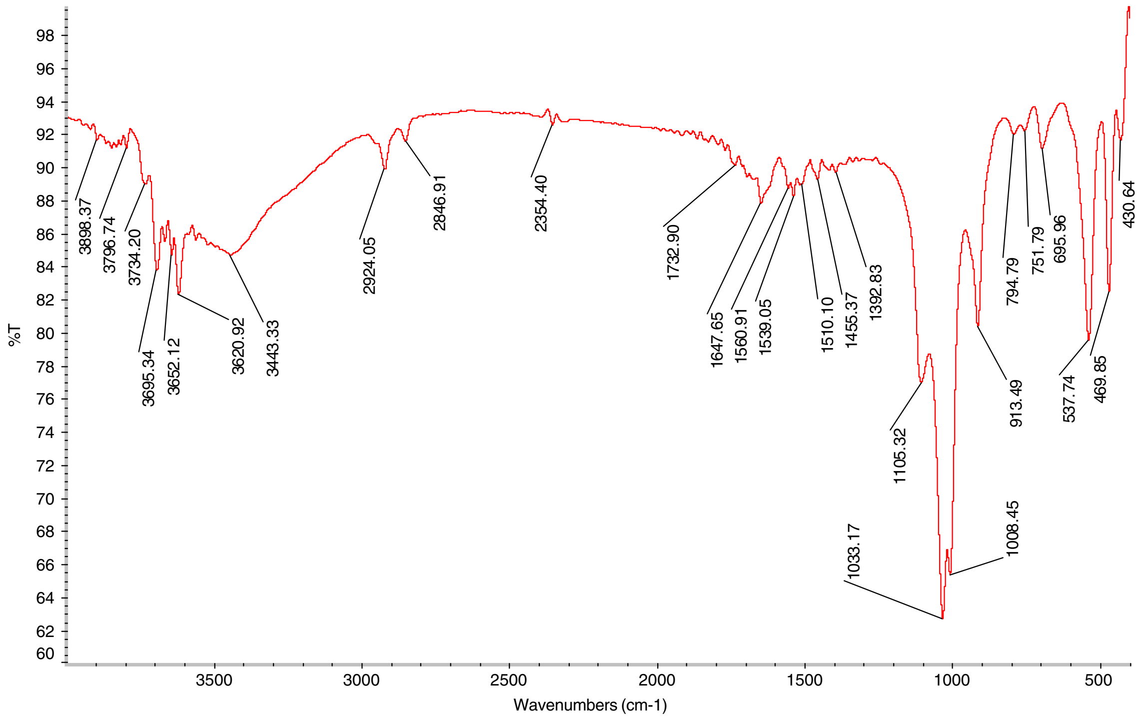

Fig. 6 FTIR spectrum of powdered brick clay (d<0.075 mm) fired at 200°C

occurrence of chemical and physical processes, for example monolayer adsorption of chromium species followed by absorption, which would fit the final outcome of the empirical approaches of the two isotherms.

According to AAS studies of the supernatant solution of Cr(VI) solution – fired brick clay suspension, metal ions such as K^+, Na^+, Mg^{2+}, Ca^{2+}, Al^{3+}, and Fe^{2+}/Fe^{3+} are detected. This indicates that while fired brick clay particles remove Cr(VI) species from solution, the above metal ions are released from the brick matrix, and hence the above ions are involved in the Cr(VI) removal process.

3.3 Mechanism of Cr(VI)—brick clay interactions

As negatively charged Cr(VI) species and negatively charged brick clay particles are not attracting to each other, it is proposed that the reduction of Cr(VI) to positively charged Cr(III) is a necessity for Coulombic attraction, in addition to specific adsorption of Cr(VI) species due to nonelectrostatic forces. Cr(VI) would be reduced by many reducing agents present is brick clay fired at 200°C, such as organic materials and minerals with surface Fe(II) species. Presence of substances containing carbonyl and hydroxyl groups in brick clay fired at 200°C is evident according to FTIR spectroscopic peaks between 1,600 and 1,700 cm^{-1}, and above 3,000 cm^{-1}, respectively (Fig. 6). Thus, there is a possibility of having substances with carboxylic acid groups in addition to surface-OH groups in the brick clay matrix. It has been reported that tartaric acid, a biological reducing agent commonly present in soils, is able to reduce Cr(VI) to Cr(III) (Deng et al. 2003), which is catalyzed by substances present in clay such as TiO_2, γ-Al_2O_3 and α-FeOOH (Deng et al. 2003; Tzou et al. 2003), supporting the claim. Reduction of Cr(VI) to Cr(III) for sorption on oxidized multi-walled carbon nanotubes, on which both Cr (III) to Cr(VI) have been detected according to X-ray photoelectron spectroscopic analysis, provides further evidence for our argument (Hu et al. 2009). When brick clay is fired beyond 200°C, organic materials undergo combustion resulting in volatile products and carbon residue, together with the loss of surface-OH groups. This is compatible with the observed mass reduction between firing temperatures of 300°C and 600°C (Fig. 2). In addition to the proposed reduction for subsequent removal, formation of carbon particles is also responsible for the adsorption of chromium species. Decrease in Cr(VI) removal ability with clay fired at higher temperatures as observed earlier (Priyantha and Bandaranayaka 2010) is attributed to the fact that reducing agents are lost due to firing. Sharp decrease in the surface area at firing temperatures above 300°C (Seneveratne and Priyantha 2009) provides another supporting evidence for the maximum removal observed with brick clay fired at 200°C.

Cr(VI) removal ability of brick clay fired at 200°C is further enhanced with acid treatment (Fig. 7). This can be explained by the facts that Cr(VI) reduction rate is increased in acidic medium and that negatively charged clay surface becomes less negative promoting the adsorption of Cr(VI) species. However, treatment of brick clay with HCl of concentration greater than 5.0 M denatures the clay as observed by visible color change, decreasing the Cr (VI) removal ability. Additionally, percentage removal of Cr(VI) neither depends on the ionic strength nor on the pH of the medium, suggesting the formation of an inner-sphere complex with brick clay fired at 200°C. Direct sorption of Cr(III) on fired brick clay also forms an inner-sphere complex as reported earlier to support these observations (Juang et al. 2003).

4 Conclusion

Treatment of Cr(VI) solution with brick clay particles of diameter <1.00 mm, fired at 200°C, in 1:10 solid/solution ratio leads to 60% removal under optimum conditions of

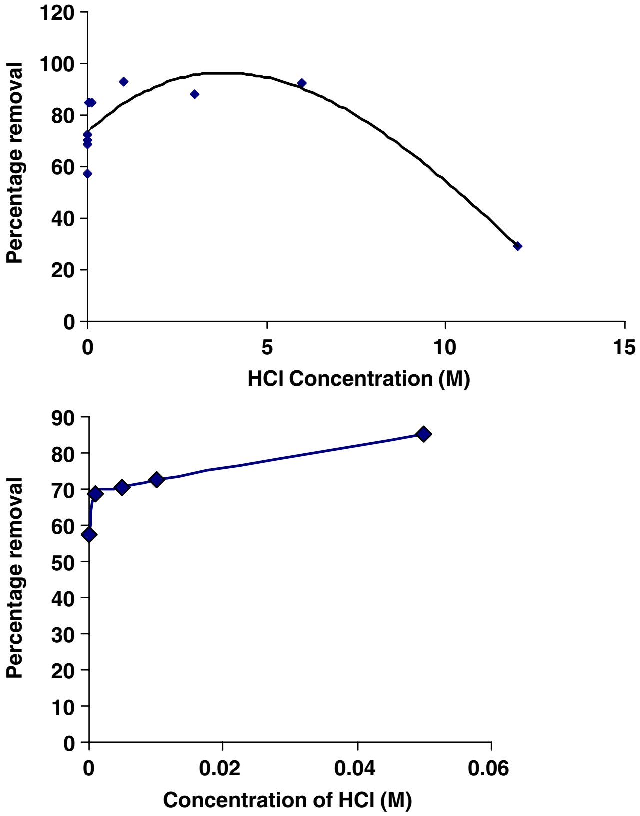

Fig. 7 Percentage removal of Cr(VI) with HCl acid-treated brick particles fired at 200°C over entire concentration range (*top*), and the effect of HCl treatment for low concentrations on an expanded scale (*bottom*) [5.0 g brick, d<1.0 mm, 50.0 cm^3 20 ppm Cr(VI) solution, 10 min stirring, 2 h settling]

10 min stirring time and 2 h settling time. The maximum removal observed at this firing temperature is probably due to the presence of reducing agents such as organic materials containing carboxylic acid functional groups and Fe^{2+} ions, which reduce Cr(VI) to Cr(III) followed by adsorption. Formation of carbon particles due to incomplete burning of organic matter is also responsible for the adsorption of both chromium species. The extent of reduction and subsequent removal of Cr(III) is enhanced when brick clay is pre-treated with acid as the reduction is facilitated by H^+ ions. Lower firing temperatures do not allow sufficient sorption sites to be exposed, while higher firing temperatures cause complete combustion of organic matter resulting in less reducing agents in brick clay. Direct sorption of Cr(VI) species by brick particles is not favorable owing to the negative charge of brick clay surfaces. Furthermore, sorption of chromium species [Cr(VI), Cr(III)] on fired brick clay obey both Langmuir and Fruendlich isotherm models, and the mechanism of Cr(VI)-brick clay interactions ultimately lead to the formation of an inner sphere complex.

Acknowledgement The authors wish to thank the National Research Council and the National Science Foundation for providing research grants (RG/2005/53 and RG/2007/FR/06, respectively). The authors also thank the Institute of Fundamental Studies, Sri Lanka, Prof. R.M.G. Rajapakse, Department of Chemistry, University of Peradeniya and Dr. Manoj Chinthaka, Department of Chemistry, University of Sri Jayawardenepura, for help provided on various aspects.

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