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Evaluation of Chitosan Modified by Sodium Dodecyl Sulfate for Removing Hexavalent Chromium from Aqueous Solutions

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Abstract: Hexavalent chromium (Cr(VI)) has the characteristic of forming anionic species, which are very toxic, very soluble in water and difficult to be removed. In this study, dichromate removal from aqueous solutions by chitosan and chitosan modified by sodium dodecyl sulfate (SDS) was addressed. The effect of various experimental parameters, such as pH (1-9), initial concentration (10-100 mg L^{-1}), adsorbent dose (0.005-0.350 g) and contact time (5-60 min) was investigated. All experiments were conducted in batch mode at room temperature (~21 ^{o}C). The obtained equilibrium adsorption isotherms were analyzed using the Langmuir and Freundlich models. Furthermore, the kinetics of dichromate removal was analyzed by pseudo-first order, pseudo-second order and the Elovich models. Optimum conditions for obtaining high removal (~97%) within a relatively short time (60 min) are: 5.0 pH, 0.100 g SDS-chitosan dosage and an initial $Cr_2O_7^{2-}$ concentration of 10 mg L^{-1}. The dichromate adsorption capacity of chitosan is 8.3 mg L^{-1}, while that of SDS-chitosan is 9.7 mg L^{-1}. In addition, the adsorption of dichromate by chitosan and SDS-chitosan is well-fitted by the Langmuir and Freundlich models while the adsorption kinetics is best fitted by the pseudo-second-order kinetic model.

Keywords: Hexavalent chromium, Adsorption, Chitosan, SDS.

Introduction

Water pollution by heavy metal ions is a serious problem which has been given attention for a long time[1]. Heavy metal ions cause health issues to human, such as damaging nerves, liver, kidney and bones, in addition to blocking the functional groups of vital enzymes[2]. Therefore, removal of heavy metal ions from aqueous solutions is considered an important process which can be achieved by different processes, such as chemical precipitation, adsorption, membrane separation and electrochemical technologies[3]. Among these processes, adsorption is, as reported in the literature, the most flexible, inexpensive and widely used one[4]. Adsorption of heavy metal ions has been accomplished using different low-cost adsor-bents, such as zeolite, coal, fly ash, clay, agricultural wastes, activated carbon, chitosan, natural oxides and industrial waste[5]. Chitosan is highly effective for removing heavy metal ions from aqueous solutions and it is the second abundant polymer after cellulose. Moreover, chitosan has several advantages, such as biocompatibility, biodegradability, nontoxicity, reactivity, film and fiber forming capacity and favorable hydrophobicity[6,7]. Therefore, chitosan is available and widely used as an absorbent in environmental remediation, including adsorption of dyes and metal ions[8].

Chitosan (Scheme 1) consists mainly of b(1-4)-linked 2-amino-2-deoxy-D-glucopyranose units and residual units of 2- acetamido-2-deoxy-D-glucopyranose[9]. Alkaline deacetylation of chitin mainly produces chitosan which can be found in some fungal cell walls[10]. Chitosan

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consists of hydrophilic functional groups and is normally insoluble in water at near-neutral pH and in most common organic solvents (e.g. DMSO, DMF, NMP, organic alcohols and pyridine)[11]. The insolubility of chitosan in aqueous and organic solvents is a result of its crystalline structure, which is attributed to extensive intramolecular and intermolecular hydrogen bonding between the chains and sheets, respectively[10]. Amino and hydroxyl groups in chitosan work as active sites and have a key role in chelating transition metal ions[12,13]. In addition, modification of chitosan by chemical and physical processes is easy and favored to improve the chitosan affinity to adsorb metal species[14]. One of the methods used to improve the chitosan surface is to use an anionic surfactant, such as sodium dodecyl sulfate (SDS) (Scheme 1)[15]. SDS is commonly used in industrial and research applications due to its low cost and excellent foaming properties[16]. Therefore, SDS was chosen to improve the adsorption capacity of chitosan for the removal of anionic Cr(VI) species from aqueous solutions, whereas positively charged chitosan formed in acidic medium reacts with negatively charged SDS to form a polyelectrolyte complex[17]. Moreover, SDS binds to chitosan surface formin bilayer or multilayer structures in

a phenomenon called 'adsolubilization'; then, heavy metal ions can be adsorbed on the chitosan-SDS surface[18].

The presence of heavy metal ions in water causes water pollution, which negatively affects human health (e.g. damage of nerves, liver, kidney and bones)[19]. Chromium ions, including Cr(III) and Cr(VI) species, are highly detected in the surface water from the effluents of industrial activites which have been classified as toxic materials to biological systems[20]. Therefore, the removal of heavy metal species from contaminated water is a significant step for environmental protection from pollution.

Removal of some transition metals, such as Cr(VI) and Cu(II), using chitosan was reported in a previous study[21], but there is no study that describes using SDS-chitosan to remove Cr(VI) from aqueous solutions. The main objectives of the current study were: 1) to attempt improving the ability of chitosan to remove hexavalent chromium from aqueous solutions using SDS, 2) to determine the optimum conditions for obtaining highest removal efficiency of hexavalent chromium from aqueous solutions and 3) to study the equilibrium and the kinetics of dichromate adsorption by both chitosan and SDS-modified chitosan.
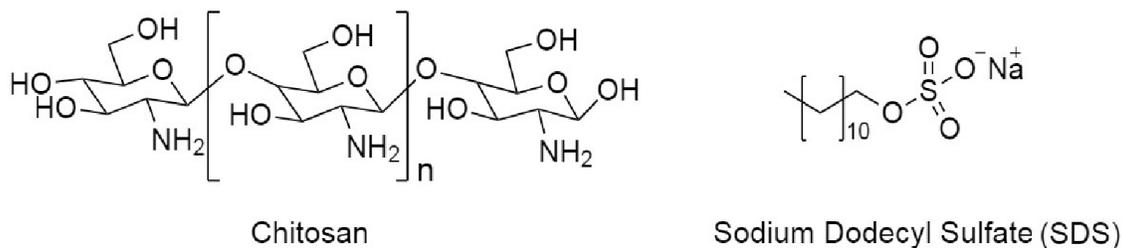
Scheme 1. Chemical structures of chitosan and sodium dodecyl sulfate (SDS).

Experimental

Materials

Chitosan (400 kDa average molecular weight and 74% degree of deacetylation), Cr(VI) from potassium dichromate (analytical grade), 1,5-diphenyl carbazide and acetone (99.0%) used in the preparation of 1,5-diphenyl carb-azide, acetic acid (99%), methanol (99.8%), hydrochloric acid (37.0%), sodium hydroxide (≥98.0%), nitric acid (65.0%) and sulfuric acid (95%). All these reagents and solvents were purchased from

Sigma Aldrich and used without further purification. Deionized water was used in the preparation of all solutions and for glassware rinsing.

Preparation of Sodium Dodecyl Sulfate (SDS)-modified Chitosan

Sodium dodecyl sulfate-modified chitosan was prepared based on a method reported in the literature[22]. In detail, an amount of 3.000 g of chitosan was dissolved in 250 mL in the presence of 7% acetic acid. The mixture was left at room temperature for 8 h, then it was added

dropwise to a coagulation mixture consisting of deionized water, methanol and sodium hydroxide (4:5:1 ratio w/w, respectively). The latter solution neutralizes with acetic acid to form spherical beads of chitosan gel. Then, chitosan beads were added to 100 mL of SDS solution (6000 mg L^{-1}) and left undisturbed at the lab bench for three days to form SDS-chitosan beads, which were filtered and washed three times with distilled water to remove the excess SDS and then dried in an oven at 50 ^{o}C for 24 h. A proposed chemical structure of SDS-chitosan was also described in reference [23].

Adsorption Experiments

All the experiments in the current study were conducted in triplicate at room temperature (~21 ^{o}C) in batch mode. In detail, 1.000 g of potassium dichromate was dissolved in 1.000 L of solution to prepare a stock solution of dichromate with a final concentration of 1,000 mg L^{-1}; then, the solution was stored in the fridge in dark. Concentrations in the range of 10 to 100 mg L^{-1} were then prepared from the stock solution by dilution. 10.0 mL of the desired dichromate solution was added to the desired chitosan or SDS-chitosan dosage (0.005-0.350 g), which is precisely weighed and loaded into a screw-capped closed vial and vigorously shaken (250 rpm). The pH of each solution in the range of 1-9 was adjusted using 0.10 M HCl or 0.10 M NaOH as measured by pH meter (pH STAT Controller MeterLab PHM 290). At desired time intervals (5-60 min), a 1.0 mL aliquot was withdrawn and analyzed using UV-VIS spectrophotometer (Systronics photometer, Model 104) at λ_{max} 540 nm. The remaining dichromate ion concentration is estimated using diphenyl carbazide as a complexing agent[24].

The removal% of Cr(VI) species was calculated using Eq. 1:

$$Removal\% = \frac{c_i - c_f}{c_i} \times 100\% \qquad (1)$$

where c_i is the initial dichromate concentration, c_f is the final dichromate concentration after adsorption by chitosan or SDS-chitosan.

Moreover, the adsorption capacity of chitosan was calculated using Eq. 2.

$$q_e = \left(\frac{c_i - c_f}{w}\right) V \qquad (2)$$

where q_e is the amount adsorbed (mg g^{-1}), c_f is the final dichromate concentration (mg L^{-1}), c_i is the initial dichromate concentration (mg L^{-1}), w

is the weight of chitosan (g) and V is the volume of solution (L).

Equilibrium Isotherms

Understanding the interaction between the adsorbate and the adsorbent can be approached by analyzing the adsorption data using various adsorption models[25]. The Langmuir and Freundlich isotherms have been tested in the current study for the adsorption experiments conducted under constant conditions of adsorbent dosage (0.100 g), contact time (60 min), pH (5.0) and initial dichromate concentration (10-100 mg L^{-1}).

Adsorption Kinetics

The adsorption kinetics was investigated at the following experimental conditions: 0.100 g adsorbent dosage, pH 5.0 and 10 mg L^{-1} initial dichromate concentration. Three common kinetic models, namely pseudo-first-order, pseudo-second-order and Elovich model, were tested to describe the adsorption kinetics.

Results and Discussion

Effect of pH

The pH of the solution significantly affected the adsorption process, whereas speciation and ionization degree of Cr(VI) and the surface of the modified chitosan are highly dependent on pH[23,26]. Figure 1 shows the effect of pH on Cr(VI) removal. Obviously, increasing the pH from 1 to 5 increases the removal% from 15% to 88% for chitosan and from 31% to 97% for SDS-chitosan. Therefore, pH 5.0 is the optimum pH to for highest removal%. Moreover, in acidic solutions (pH 1–6), chromium ions can be found in different forms; $Cr_2O_7^-$, $HCrO_4^-$, $Cr_3O_{10}^{2-}$ and $Cr_4O_{13}^{2-}$, considering $HCrO_4^-$ ions as predominant. Therefore, the high removal% of Cr(VI) species at pH 5.0 is due to the strong electrostatic interactions between $HCrO_4^-$ and the protonated amino groups in chitosan[27]. With increasing pH of the solution above 6.4, the CrO_4^{2-} and $Cr_2O_7^{2-}$ ions become the predominant ions in the solution and their interactions with the deprotonated chitosan surface are less, in addition to the presence of competing hydroxyl ions; therefore, the removal% of Cr(VI) species decreases[28]. In addition, methylene groups in SDS may increase the surface area available for adsorption when SDS connects with chitosan[23], which explains the higher removal% in case of
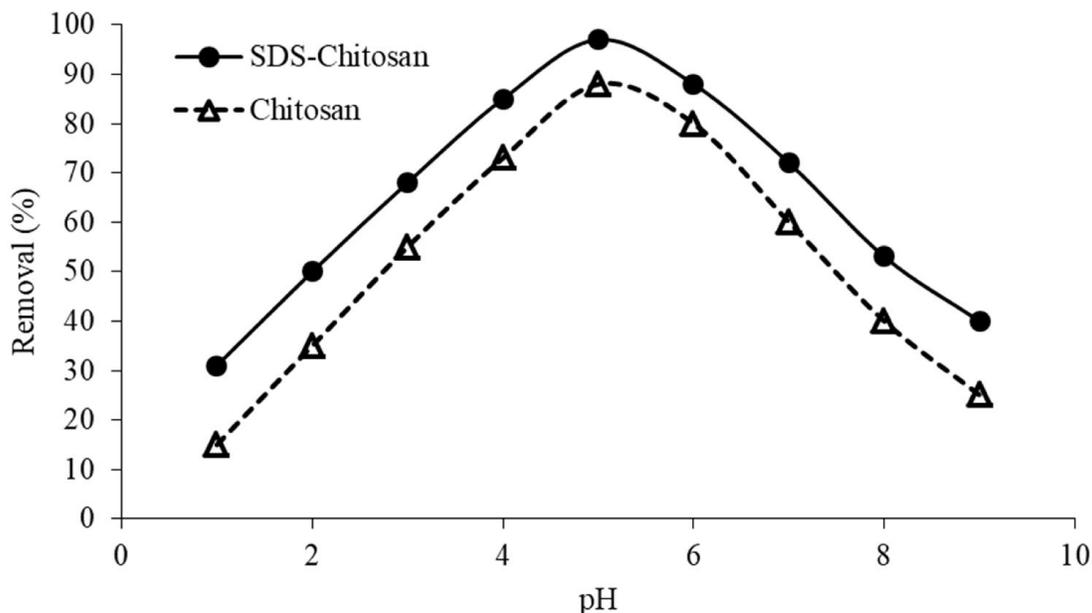
SDS-chitosan compared to unmodified chitosan.

Figure 1. Effect of pH on the removal of Cr(VI) from aqueous solution using chitosan and SDS-chitosan: adsorbent dosage = 0.100 g, c_i = 10 mg L^{-1} and t = 60 min. (n = 3, RSD < 10%).

Effect of Adsorbent Dosage

Adsorbent dosage has a clear effect on Cr(VI) removal, as shown in Figure 2. Increasing the amount of chitosan or SDS-chitosan from 0.005 g to 0.100 g increases the removal% from 44% to 97% using SDS-chitosan and from 27% to 87% using chitosan. This behaviour is due to the increase in the surface area and the number of active and available sites to adsorb Cr(VI) ions[29]. In contrary, the amount adsorbed decreases with increasing the adsorbent dosage (notice Eq. 2). Therefore, the optimum adsorbent dosage of SDS-chitosan or chitosan to obtain the maximum removal of Cr(VI) is 0.100 g.

Effect of Initial Concentration

Figure 3 shows the effect of initial concentration on the removal% of Cr(VI). Clearly, the removal% decreases with increasing the initial concentration of Cr(VI) from 10 to 100 mg L^{-1}. This behaviour is due to larger ratio between active sites on adsorbent surface to Cr(VI) ions at low concentration (10 mg L^{-1}) compared to smaller ratio at large concentration (100 mg L^{-1})[30]. Nevertheless, the amount adsorbed increases from 0.89 mg g^{-1} to 6.1 mg g^{-1} of chitosan and from 0.98 mg g^{-1} to 7.0 mg g^{-1} of SDS-chitosan with increasing the Cr(VI)

concentration from 10 mg L^{-1} to 100 mg L^{-1}.

Effect of Contact Time

Figure 4 shows that Cr(VI) removal increases with increased contact time until a constant value is reached at 60 min representing equilibrium (~95% removal by SDS-chitosan and ~84% by chitosan). Moreover, the adsorption on chitosan and SDS-chitosan is fast in the beginning due to the large number of available sites on the adsorbent surface[31].

Adsorption Isotherms

Langmuir and Freundlich isotherm models are widely used to describe the adsorption of ionic species[32]. The Langmuir model is represented by Eq. 3[33]:

$$\frac{c_e}{q_e} = \frac{1}{Q_o b} + \frac{c_e}{Q_o} \qquad (3)$$

where c_e is the equilibrium concentration (mg L^{-1}), q_e is the amount adsorbed at equilibrium (mg g^{-1}), Q_0 is the adsorption capacity (mg g^{-1}) and b is the equilibrium adsorption constant (L mg^{-1}). The adsorption process is well fitted by the Langmuir isotherm (Figure 5A ; R^2 is 0.9894 for SDS-chitosan and 0.9994 for chitosan) ; the values of Q_0 and b were calculated and are listed in Table 1.

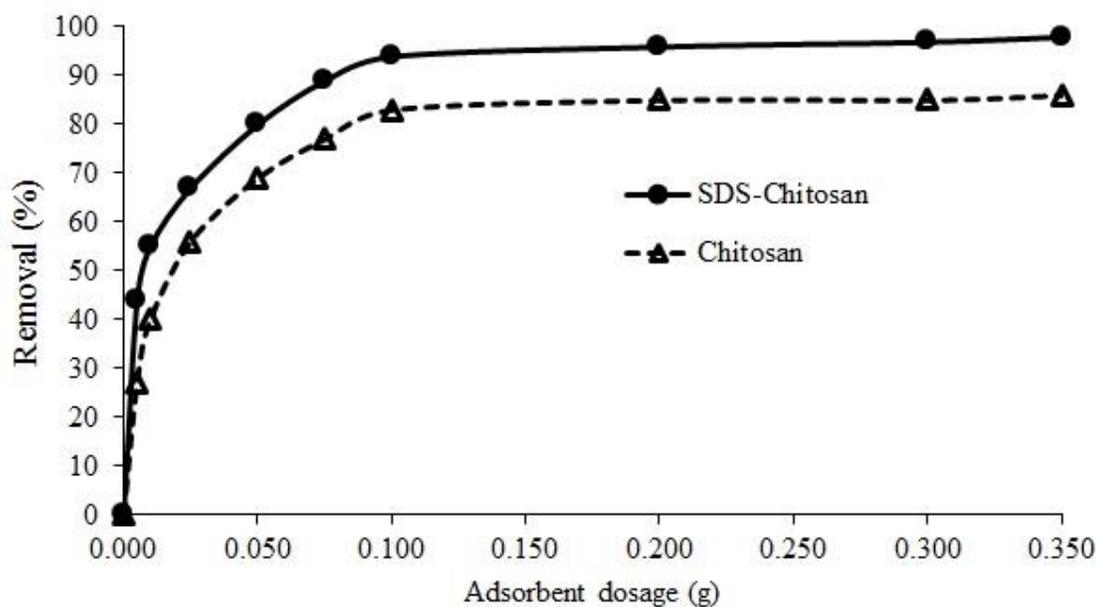**Figure 2. Effect of chitosan and SDS-chitosan dosage on Cr(VI) removal. Experimental conditions: pH = 5.0, c_i = 10 mg L^{-1} and t = 60 min. (n = 3, RSD < 7%).**
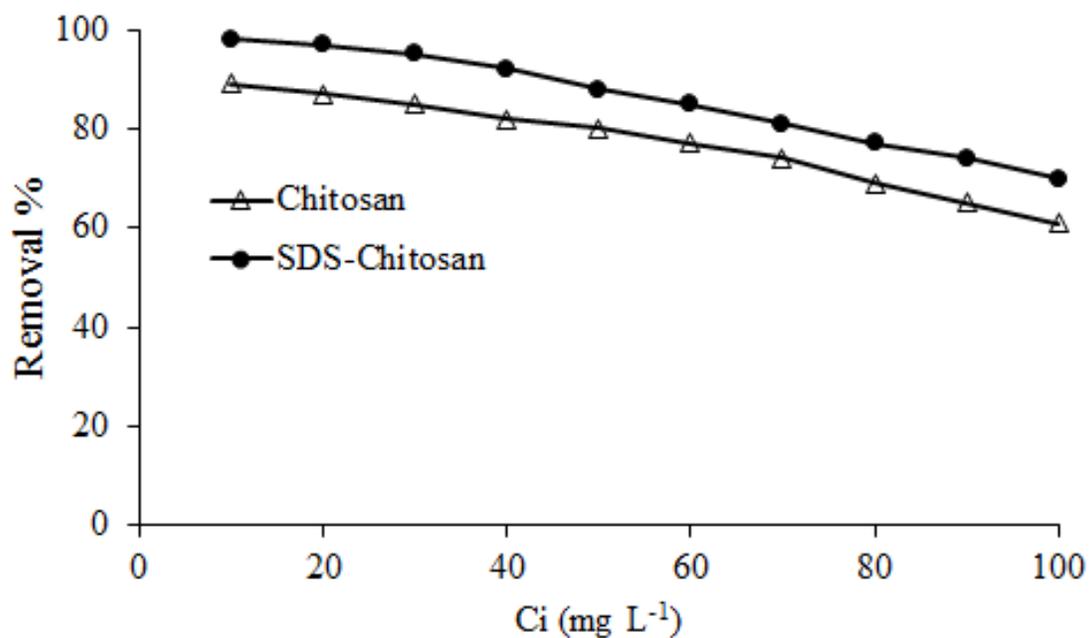

Figure 3. Effect of initial concentration on Cr(VI) removal using chitosan and SDS-chitosan. Experimental conditions: pH = 5.0, adsorbent dosage = 0.100 g and t = 60 min. (n = 3, RSD < 5%).
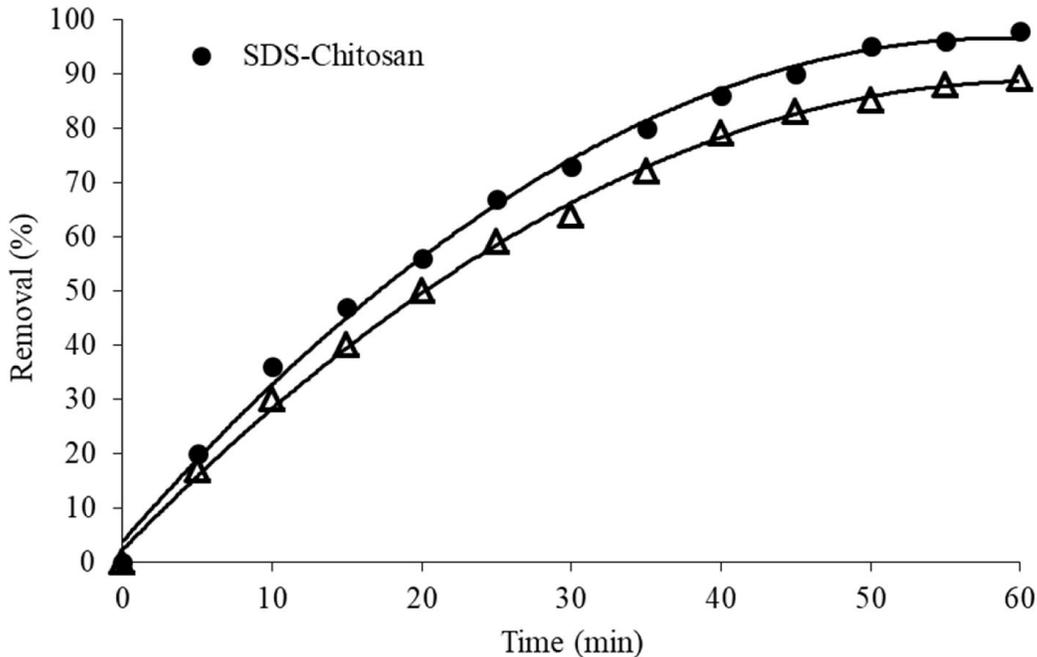
Figure 4. Cr(VI) Removal% by chitosan and SDS-chitosan as a function of time. Experimental conditions: pH = 5.0, c_i = 10 mg L^{-1} and adsorbent dosage = 0.100 g. (n = 3, RSD < 9%).

The Freundlich isotherm is represented by Eq. 4:

$$\ln q_e = \ln K_f + 1/n \times \ln C_{eq} \qquad (4)$$

where q_e is the amount adsorbed (mg g^{-1}), c_{eq} is the equilibrium concentration in solution (mg L^{-1}), $1/n$ is an index to adsorption intensity and K_f is the Freundlich constant related to the adsorption capacity (g L^{-1}). The Freundlich plot is shown in Figure 5B and the values of $1/n$ and K_f are listed for chitosan and SDS-chitosan in Table 1. The adsorption of Cr(VI) species is well fitted by the Freundlich model (R^2 is 0.9959 for chitosan and 0.9992 for SDS-chitosan) ; the K_f values are 0.3987 mg g^{-1} for chitosan and 0.4810 mg g^{-1} for SDS-chitosan. Therefore, SDS-chitosan has a higher ability to adsorp Cr(VI)

from aqueous solutions than chitosan.

The adsorption capacity of SDS-chitosan used in the current study (~7.4 mg g^{-1}) is higher than those of other adsorbents, such as activated alumina (1.6 mg g^{-1}), modified oak sawdust (1.7 mg g^{-1})[34] and cross-linked chitosan (6 mg g^{-1})[35]. In contrary, some adsorbents, such as bismuth impregnated biochar (0.1 g adsorbent, 50 mL of 50 mg L^{-1} adsorbate, pH 3.0 and 24h contact time), had adsorption capacity of 12.23 mg g^{-1}[36] and carbon nano-onions (1.5 mg adsorbent, 12 mL of 25 mg L^{-1} adsorbate, pH 3.4 and 24 h contact time) had 23.53 mg g^{-1}[37]; these adsorbents show a higher adsorption capacity for Cr(VI) adsorption from aqueous solutions.

Table 1. Langmuir and Freundlich isotherm parameters for Cr(VI) removal by chitosan and SDS-chitosan.

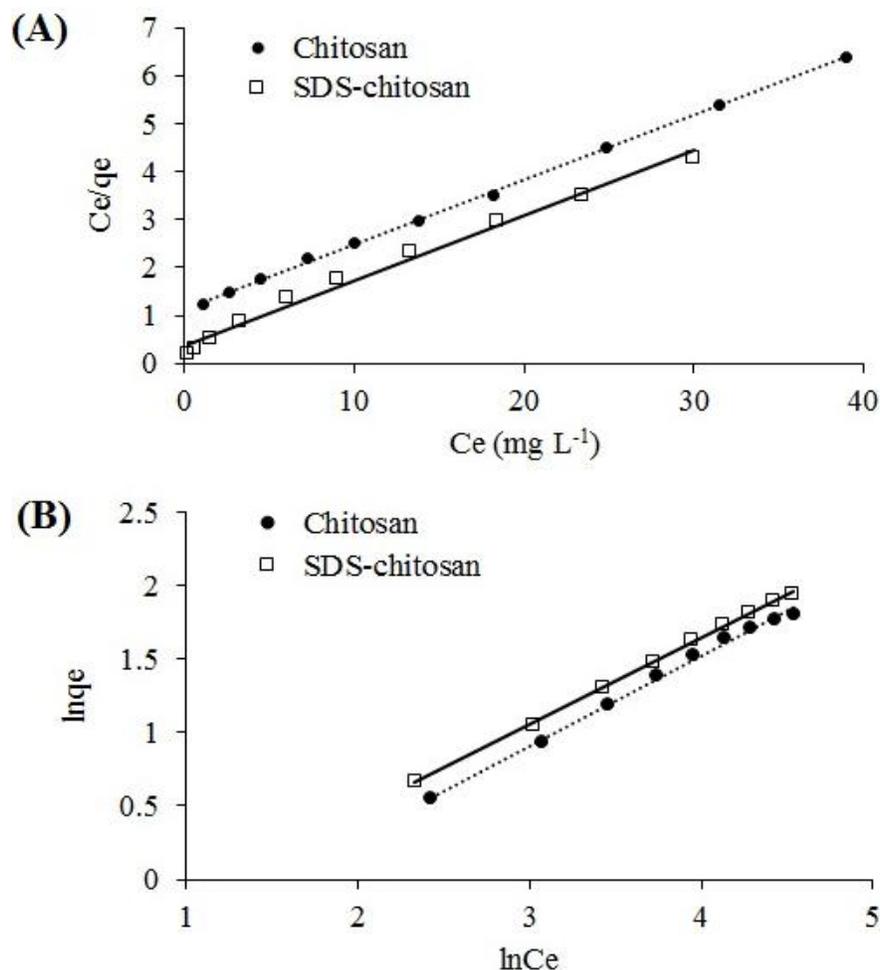| Model	Parameter	Chitosan	SDS-chitosan
Langmuir	Q_0 (mg g^{-1})	7.35	7.43
	b (L mg^{-1})	0.118	0.357
	R^2	0.9994	0.9894
Frendluich	K_f (mg g^{-1})	0.3987	0.4810
	1/n	0. 6109	0.5950
	R^2	0.9959	0.9992

Figure 5. Linearized form of Langmuir (A) and the Freundlich (B) adsorption isotherms for Cr(VI) removal by chitosan and SDS-chitosan.

Kinetic Analysis

The experimental data was analyzed using pseudo-first-order (Eq. (5)), pseudo-second-order (Eq. (6)) and the Elovich kinetic model (Eq. (7))[38], as shown in Figure 6.

$$log\ (q_e - q_t) = log\ q_e - (k_1/2.303)t \qquad (5)$$

$$\frac{t}{q_t} = \frac{1}{k_2\ q_e^2} + \frac{1}{q_e}t \qquad (6)$$

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \qquad (7)$$

where k_1 is the rate constant of adsorption (min^{-1}), k_2 is the rate constant of adsorption (mg g^{-1} min^{-1}), q_e is the amount adsorbed at equilibrium (mg g^{-1}), q_t *is* the amount adsorbed at any time t (mg g^{-1}), α is the initial adsorption rate (mg g^{-1} min^{-1}) and β is the desorption constant (g mg^{-1}).

Values of the related terms were calculated and are listed in Table 2. These values indicate that Cr(VI) adsorption by chitosan and SDS-chitosan is best fitted to the pseudo-second-order model (R^2 of chitosan = 0.9915 and R^2 of SDS-chitosan = 0.9961). In addition, the initial adsorption rate (α) of chitosan is 0.08751 mg g^{-1} min^{-1} and is 0.08893 mg g^{-1} min^{-1} for SDS-chitosan. Therefore, chemisorption is probably the determining step of the adsorption rate of Cr(VI) by chitosan and SDS-chitosan[39]. Moreover, several studies indicated that Cr(VI) removal by an adsorbent is fitted to pseudo-second-order model[40–43]. The rate constants (k_1 and k_2 in Table 2) are slightly higher for SDS-chitosan compared to chitosan, which can be attributed to the increase of the average size of SDS-chitosan particles[44].
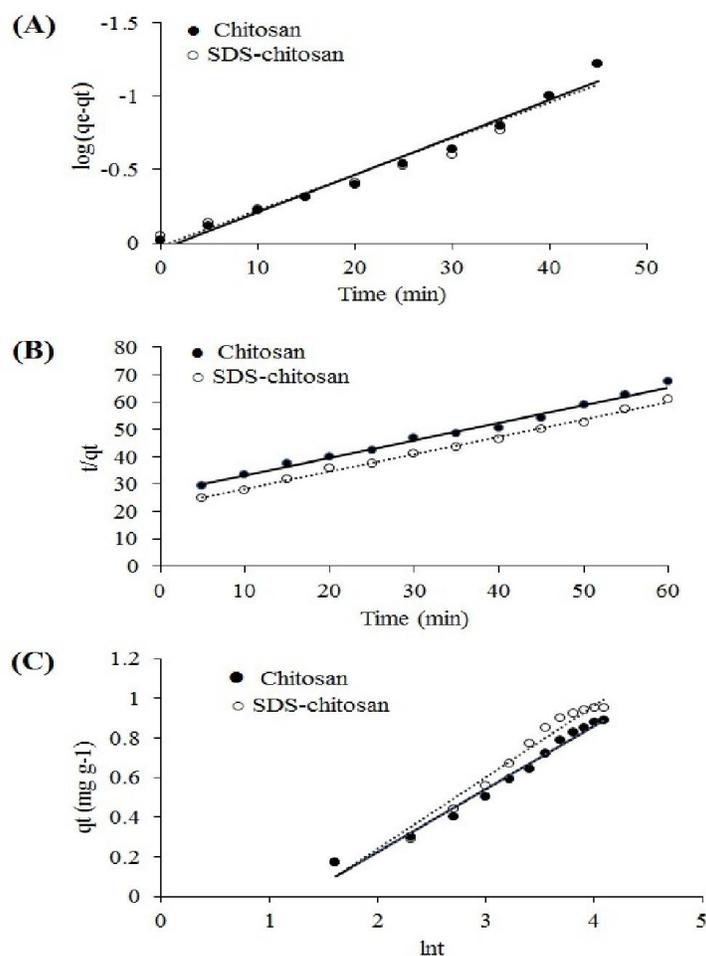
Figure 6. Kinetic study models for Cr(VI) removal using chitosan and SDS-chitosan: Pseudo-first-order model (A), pseudo-second-order model (B) and Elovich model (C).

Table 2. Kinetic parameters using pseudo-first-order model, pseudo-second-order model and Elvoich model for Cr(VI) removal by chitosan and SDS-chitosan.

Pseudo-first-order model			
Adsorbent	k_1 **(min^{-1})**	q_e **(mg g^{-1})**	R^2
Chitosan	0.05942	1.1657	0.9562
SDS-chitosan	0.06103	1.2039	0.9710

Pseudo-second-order model			
	k_2 **(g mg^{-1} min^{-1})**	q_e **(mg g^{-1})**	R^2
Chitosan	0.01583	1.5420	0.9915
SDS-chitosan	0.01859	1.5701	0.9961

Elovich model			
	β **(g mg^{-1})**	α **(mg g^{-1} min^{-1})**	R^2
Chitosan	3.1556	0.08751	0.9759
SDS-chitosan	2.7793	0.08893	0.9737

Conclusion

Modification by SDS introduces only a slight enhancement by $\sim 10\%$ in the ability of chitosan to remove Cr(VI) from aqueous solutions. The removal of Cr(VI) by chitosan and SDS-chitosan could be successfully described by the Langmuir and Freundlich isotherm models and the adsorption kinetics is best fitted by the pseudo-second-order model. Further studies related to desorption of Cr(VI) from chitosan and SDS-chitosan will be a useful strategy to understand the strength of binding between the adsorbent and the Cr(VI) species, in addition to evaluating the reusability of chitosan and SDS-chitosan.

Conflict of interest

The author does not have any conflict of interest.

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