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ORIGINAL ARTICLE

Fast and selective adsorption of As(V) on prepared modified cellulose containing Cu(II) moieties

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Abstract Arsenic is one of the major pollutants and a worldwide concern because of its toxicity and chronic effects on human health. The synthesis of copper containing modified cellulose (Cell-N-Cu) adsorbent was achieved. The adsorbent was used to purify different waters from As(V) at different experimental conditions using batch method. Kinetic and thermodynamic properties as well as the mechanism of interaction between As(V) and adsorbent active sites were discussed. The adsorption behavior of the obtained samples showed high adsorption capacity as well as fast kinetics towards As(V) in their solutions. The maximum adsorption capacities of As(V) on Cell-N-Cu adsorbent were found to be 1.32 mmol/g. Regeneration and durability of the loaded adsorbent towards the successive reuse were also investigated. Fast and selective separation of As(V) from a real wastewater sample indicated that the investigated resin is potentially attractive for arsenic removal from different waste streams.

1. Introduction

Arsenic is toxic and its presence in drinking water causes toxic and carcinogenic effects on human beings. It is reported that long-term drinking of arsenic contaminated water causes gastrointestinal, skin, liver, and nerve tissue injuries (Saha et al., 1999; ATSDR, 2002).

United States Environmental Protection Agency (USEPA) in 2001 reduced the limit of arsenic in drinking water to 10 mg/L from an earlier value of 50 mg/L (Zaspalis et al., 2007; Kundu and Gupta, 2007; Pagana et al., 2008). During the last 20 years, naturally occurring arsenic was found to be widespread in natural water in the USA, Argentina, Taiwan, China, Hungary, Vietnam, Chile, Bangladesh and India. It was estimated that some 35–77 million people were at extreme risk of groundwater arsenic poisoning in Bangladesh (Chakravarty et al., 2002; Souter et al., 2003; Singh et al., 2004; Kundu and Gupta, 2005).

Arsenic exists in natural waters in both inorganic as well as organic forms. The inorganic form of arsenic is more toxic than its organic form. Inorganic arsenic exists in natural waters in two oxidation states, As(III) and As(V). As(III) is dominant in more reduced conditions whereas As(V) is

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dominant in an oxidizing environment (Xu et al., 1991; Anawar et al., 2003; Ng et al., 2003). Several technologies have been employed to remove arsenic from water, including reverse osmosis, electrodialysis, coagulation, ion exchange, adsorption, and chemical precipitation. Adsorption is an economical and a reliable technique which is considered as an efficient alternative for removal of arsenic (Aredes et al., 2012). Different adsorbents such as activated carbons (Lorenzen et al., 1995), biological materials (Oehmen et al., 2006; Halttunen et al., 2007; Devi et al., 2008; Mondal et al., 2008), mineral oxides (Cumbal et al., 2003; Sun et al., 2006) were applied. Metal-loaded resins were also successfully used for arsenic adsorption. Metal-loaded chelating resins have advantages of large binding energy, good stability, high preconcentration factor, mechanical stability and good reproducibility. Iron(III)-containing resins and Zr(IV)-loaded chelating resin were studied for the removal of As(V) and As(III) from aqueous solutions (Matsunga et al., 1996; Haron et al., 1999; Balaji et al., 2005). In our group of research, we earlier reported on synthetic resins containing Cu(II) moieties loaded on GMA/MBA-TEPA resin and discussed its adsorption behavior towards As(V) (Donia et al., 2013).

In this study, renewable, inexpensive and eco-friendly copper containing biosorbent (Cell-N-Cu) has been successfully utilized for adsorption of As(V) from aqueous medium, with high adsorption capacity as well as fast uptake. The uptake characteristics have been studied using batch method at different experimental conditions. Kinetic and thermodynamic parameters of the uptake process were also calculated and discussed. The regeneration of the loaded resin for the repeated use was investigated.

2. Experimental

2.1. Chemicals

Cellulose (Cell), phosphorous oxychloride ($POCl_3$), dimethylformamide (DMF), and tetraethylenepentamine (TEPA) were Aldrich products. All other chemicals were Prolabo products and used as received. Copper acetate [$Cu(CH_3COO)_2$] and sodium arsenate [$Na_2HAsO_4 \cdot 7H_2O$] were used as a source of Cu(II) and As(V), respectively.

2.2. Preparation of cellulose adsorbent

2.2.1. Preparation of chlorodeoxy cellulose (Cell-Cl)

A sample of 10 g of Cell was suspended in 200 mL DMF for one hour then reacted with 6 mL of $POCl_3$ under mechanical stirring for more than 15 min. The cellulose chloride (Cell-Cl) obtained was filtered off then washed with DMF, distilled water and 5% NaOH followed by distilled water again and then dried in air for 24 h. (Gennaro et al., 1983).

2.2.2. Preparation of amine chelating resin (Cell-N)

A sample of 1.0 g Cell-Cl was directly reacted with 6 mL of TEPA without using any solvent. The reaction mixture was stirred under heating for 3 h at 130 °C. The product obtained was filtered off, washed with distilled water followed by methanol and then dried in air (Da Silva Filho et al., 2006). Synthetic route followed to obtain Cell-Cl and Cell-N from cellulose was represented in Scheme 1.

2.2.3. Estimation of amino group content

The concentration of amino group of Cell-N was estimated using a volumetric method as follows (Atia et al., 2008). A 40 mL of 0.05 M HCl solution was added to 0.1 g of Cell-N and conditioned for 15 h on a Vibromatic-384 Shaker. The residual concentration of HCl was estimated through the titration against 0.05 M NaOH solution and phenolphthalein as indicator. The number of moles of HCl interacted with amine groups and consequently the amine group concentration (mmol/g) was calculated from the following equation

$$\text{Concentration of amino groups} = \frac{(M_1 - M_2) \times 40}{0.1} (\text{mmol/g of resin}) \tag{1}$$

where M_1 and M_2 are the initial and final concentrations of HCl, respectively.

2.2.4. Loading of Cell-N adsorbent by Cu(II)

A 0.1 g of Cell-N adsorbent was stirring with 100 mL of 1×10^{-2} M copper acetate solution on a Vibromatic shaker at 300 rpm at room temperature and natural pH for 2 h. The adsorbed amount was calculated from the residual concentration of Cu(II) in the equilibrated solutions via titration against 5×10^{-3} M EDTA using murexide as indicator (Atia et al., 2003). The product obtained was filtered off, washed with distilled water and then dried in air and named Cell-N-Cu.

2.2.5. Characterization of the adsorbent

2.2.5.1. SEM–EDX analysis. The morphology of sorbent particles and the EDX analysis were determined using an FESEM–EDX system (Quanta FEG 200, equipped with an OXFORD Inca 350 Energy Dispersive X-ray microanalysis (EDX) system) at an accelerating voltage of 30 keV and a working distance of 10 μm.

2.3. Adsorption measurements using batch method

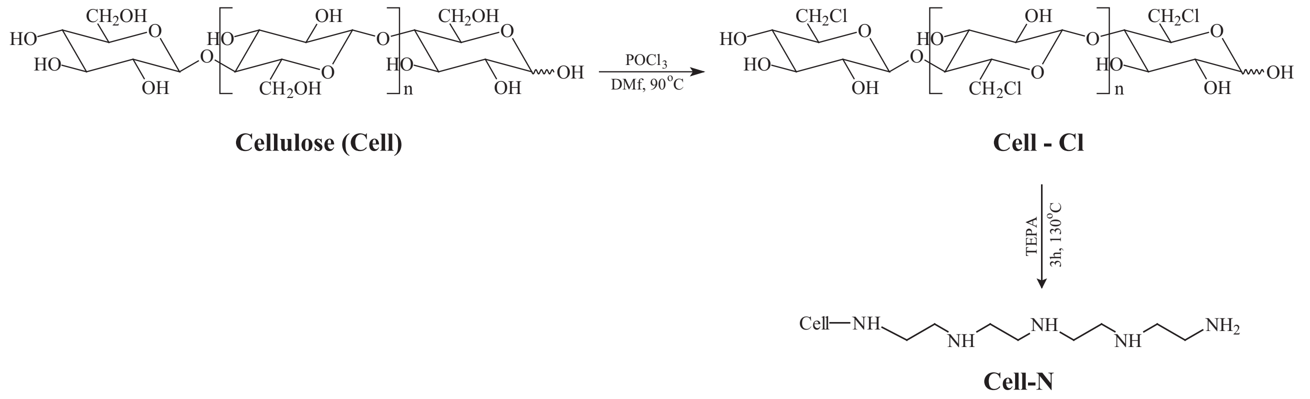
2.3.1. Preparation of solutions and concentration measurements

Stock solution (1×10^{-2} M) of As(V) was prepared from sodium arsenate in distilled water. The desired concentrations were then obtained by dilution. The concentration of As(V) was measured using the molybdene blue spectrophotometric method (Lenoble et al., 2003). Calibration curve of As(V) in distilled water was constructed from the absorbance against concentration of As(V) at wave length of 870 nm. The measurements were carried out using double beam UV/visible spectrophotometer, CECIL CE 7400, Aquarius. The path cell length was 1.0 cm. Each measurements reading was an average of three replicates.

2.3.2. Effect of initial pH

A 0.1 g portions of the adsorbent was placed in a series of flasks each contained 100 mL of 1×10^{-3} M As(V) solutions. The desired pH solutions were adjusted using 0.1 N HCl or 0.1 N NaOH. The flasks were conditioned on a Vibromatic shaker at 300 rpm for 1 h at 25 °C. The uptake of As(V) was calculated by determining the residual concentration of As(V) following the above method and according to equation

$$q_e = \frac{(C_o - C_e) \times 100}{0.1} \tag{2}$$



Scheme 1 Synthetic route followed to obtain Cell-Cl and Cell-N from cellulose.

where q_e is the uptake of As(V) at equilibrium, C_o and C_e are the initial and equilibrium concentrations of As(V), respectively.

2.3.3. Effect of contact time

A 0.1 g portions of the adsorbent was placed in flasks containing 100 mL of 1×10^{-3} M As(V) at natural pH 8.4 (natural). The contents of the flasks were equilibrated on the shaker at 300 rpm and 25 °C. Two mL of the solution (free from adsorbent particles) was taken from the flasks at different time intervals and the residual concentrations of As(V) were determined.

2.3.4. Adsorption isotherms

The effect of initial concentration of As(V) on the adsorption was carried out by placing 0.1 g portions of the adsorbent in a series of flasks each contains 100 mL of As(V) with different concentrations at pH 8.4 (natural). The contents of the flasks were equilibrated on the shaker at 300 rpm and at 25, 35, 45 and 55 °C for 15 min. After equilibration, the residual concentration of As(V) in each flask was determined to calculate the amount adsorbed of As(V).

2.4. Regeneration of the loaded adsorbent

Regeneration experiments were performed by placing 0.1 g of the adsorbent with 100 mL of 1×10^{-3} M As(V) solutions at suitable pH. The solution was decanted and the adsorbent was washed by distilled water. The total uptake was estimated. The loaded adsorbent was then subjected for elution using ammonium hydroxide solution (1.6 M). The adsorbent was then carefully washed with distilled water to become ready for reuse in the second uptake run. The regeneration efficiency was calculated according to equation

$$\text{Regeneration efficiency } (\%) = \frac{\text{Uptake in the 2nd run}}{\text{Uptake in the 1st run}} \times 100 \tag{3}$$

2.5. Selective adsorption of As(V) from a real wastewater sample

To minimize arsenic hazards and get cleaner wastewater, a 100 mL wastewater real sample obtained from a water treatment plant was conditioned with 0.1 g of Cell-N-Cu adsorbent till equilibrium time at natural pH and 28 °C. The concentrations of As(V) were analyzed before and after treatment using inductively coupled plasma (ICP) following the above method.

3. Results and discussion

3.1. Adsorbent characterization

The synthesis of modified cellulose containing copper (II) moieties was carried out by loading Cell-N that contains amine

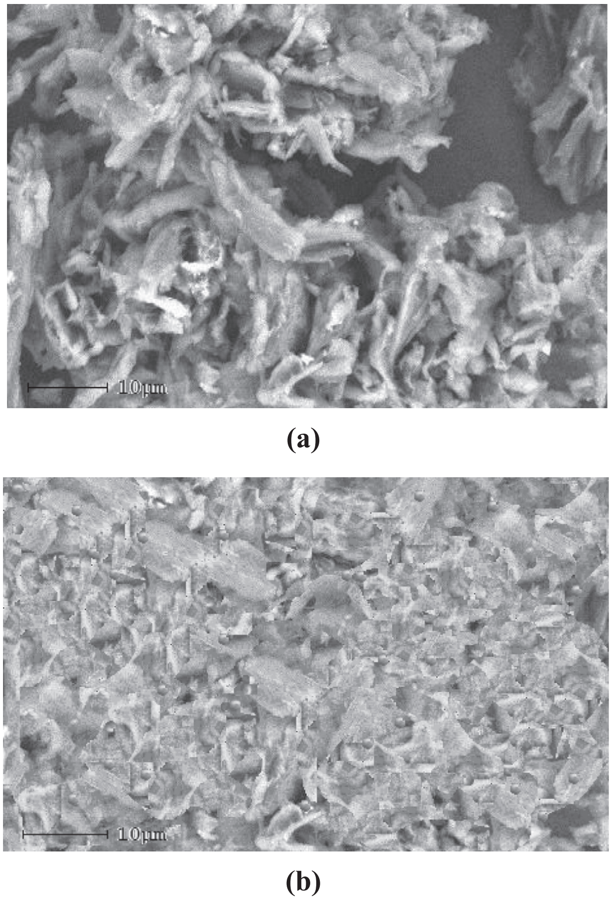
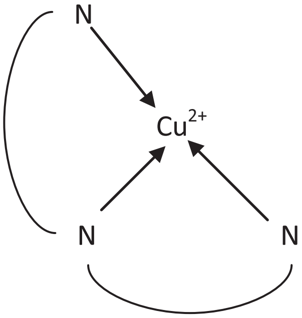


Figure 1 Scanning electron micrographs of Cell-N-Cu before (a) and after (b) loading with As(V).



Scheme 2 The coordination mode of Cu(II) moieties with amino groups in Cell-N-Cu adsorbent.

groups (estimated value of amine groups was 4.8 mmol/g) by Cu(II). The presence of amine modified cellulose (Cell-N) was confirmed by FT-IR spectra and TGA measurements (Donia et al., 2013).

The morphology of Cell-N-Cu before and after loading with arsenic was examined using scanning electron microscopy (SEM) (Fig. 1a and b). The SEM micrograph of Cell-N-Cu showed a coarse surface with several small pores inside and suggests interconnected pores within the resin (Fig. 1a). However, an apparent difference in the surface morphology of the sorbent after As(V) adsorption was observed (Fig. 1b). The surface roughness was decreased. Some of formerly existing pores disappeared and small particles were found to adhere to the surface of the sorbent. These results partially demonstrated that the chemical interactions took place between As(V) ions and Cell-N-Cu.

The success of treatment and presence of copper in Cell-N-Cu was confirmed using titration against 5×10^{-3} M EDTA (from which the loaded Cu(II) was found to be 1.58 mmol/g). Quantitative value of loaded Cu(II) was slightly equal to one third of the concentration of the amine groups (4.8 mmol/g). Therefore, Cu(II) may be coordinated with three amine groups as illustrated in Scheme 2.

To map elemental copper and arsenic qualitatively, EDX measurements were performed and Cell-N-Cu showed distinct signals at 0.9 and 8.0 keV corresponding to copper while As(V) loaded Cell-N-Cu showed distinct signals at 1.3 and 10.5 keV corresponding to Arsenic (Fig. 2).

3.2. Adsorption studies

3.2.1. Effect of initial pH

The adsorption of As(V) on Cell-N-Cu adsorbent as a function of pH was shown in Fig. 3 using batch process. The highest uptake value was observed at pH 8.4 (natural) and reached 0.82 mmol/g. This may be related to the presence of As(V) as $HAsO_4^{2-}$ species which has a good capability to interact with positively charged Cu(II) on the adsorbent (strong Lewis base species) (Gupta and Chen, 1978; Fryxell et al., 1999). The lower adsorbed amount of As(V) compared to the estimated quantitative amount of Cu(II) present in Cell-N-Cu may be related to the bulky size of $HAsO_4^{2-}$ group which leads to insufficient saturation of active sites.

The decrease in the uptake value was observed with lower pH may be attributed to the presence of As(V) as $H_2AsO_4^-$ species (with relative lower ability for interaction with Cu(II)) (Ghosh and Yuan, 1987; Ramana and Sengupta, 1992). On the other hand, when the pH of solution is above 9, the competitive adsorption of hydroxyl ions with arsenic ions $HAsO_4^{2-}$ in solution causes a decrease in uptake of arsenic on Cell-N-Cu adsorbent (An et al., 2005).

3.2.2. Kinetic studies

The adsorption of As(V) by Cell-N-Cu adsorbent as a function of time was shown in Fig. 4. The indicated figure showed that the adsorption was initially rapid, and was followed by a slower removal that gradually approaches equilibrium: about 60% of arsenic was removed within the first minute of contact. The rapid adsorption in the beginning stage can be attributed to the greater concentration gradient and more available sites for adsorption. The maximum uptake capacity was achieved

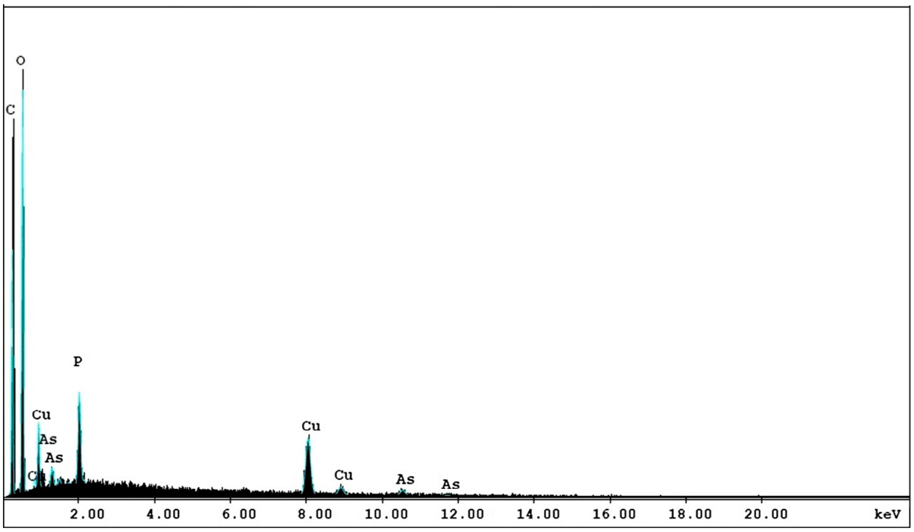


Figure 2 EDX spectra of Cu(II) and As(V) on loaded Cell-N-Cu adsorbent.

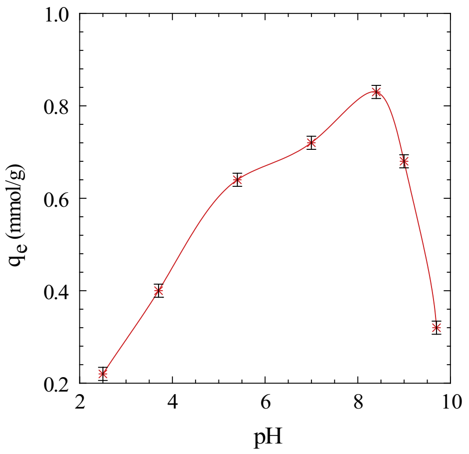


Figure 3 Effect of pH on the adsorption of As(V) by Cell-N-Cu adsorbent using initial concentrations of 1×10^{-3} M, 1 h equilibrium time and 25 °C.

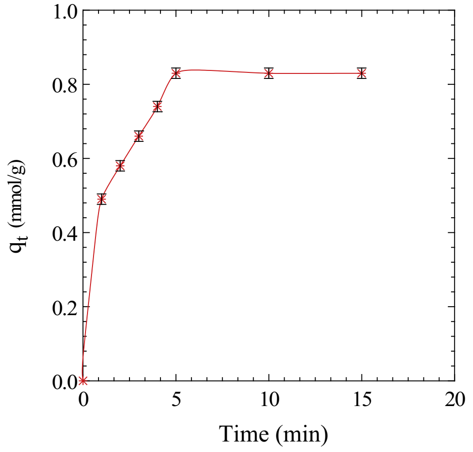


Figure 4 Effect of time on the adsorption of As(V) by Cell-N-Cu adsorbent using initial concentrations of 1×10^{-3} M and 25 °C.

within five minutes. This too fast rate of adsorption makes this adsorbent promising for practical applications if compared with other reported sorbents (Table 1) (Gupta and Babu, 2009; Martinson and Reddy, 2009; Zhang et al., 2013). This may be attributed to the hydrophilic as well as the swelling characters of cellulose.

The adsorption/time data obtained in Fig. 4 were applied to two simplified kinetic models, including pseudo-first-order model and pseudo-second-order model. The linear pseudo-first-order model may be expressed as (Shin et al., 2011).

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \tag{4}$$

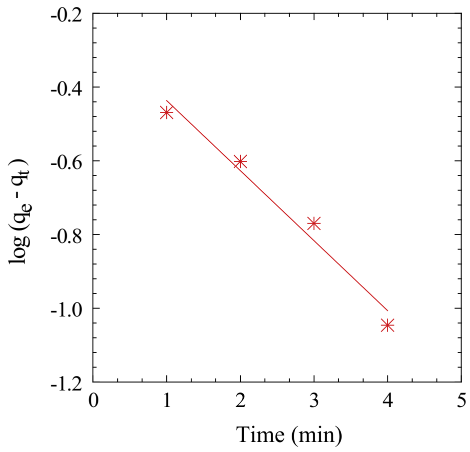


Figure 5 Pseudo-first order plots for the adsorption of As(V) on Cell-N-Cu adsorbent.

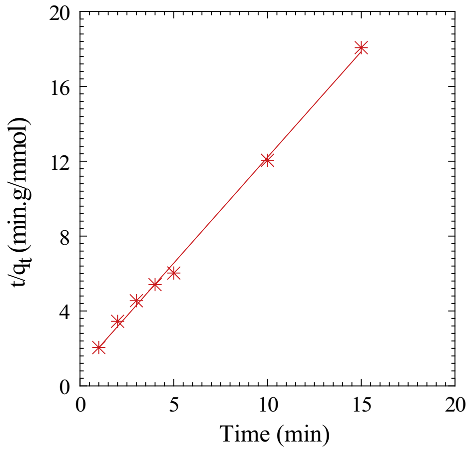


Figure 6 Pseudo-second order plots for the adsorption of As(V) on Cell-N-Cu adsorbent.

where k_1 is the pseudo-first order overall rate constant (min^{-1}), q_e and q_t refer to the amount of As(V) adsorbed at equilibrium and at time t, respectively (mmol/g).

The linear pseudo-second order kinetic model was expressed as (Hoda et al., 2006)

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right)t \tag{5}$$

where k_2 is the overall rate constant of the pseudo-second order adsorption (g/mmol min). The validity of the models was checked by calculating the correlation coefficient values of the straight lines (R^2) as well as by comparing values of experimental and calculated values of q_e. The adsorption

Table 1 Comparison of adsorption capacities for As(V) using various adsorbents.

Adsorbent	Initial pH	Time	Conc. range (mg/L)	Adsorption capacity (mg/g)	References
Fe(III)–Ti(IV) oxide	7.0	6 h	5.0–250	14.3	Gupta and Ghosh (2009)
CuO nanoparticles	8.0	3 h	0–100	22.6	Martinson and Reddy (2009)
Fe–Cu binaryoxide	7.0	24 h	0–60	82.7	Zhang et al. (2013)
Cell-N-Cu	8.4	5 min	100–700	98.9	This work

Table 2 Pseudo-second and pseudo-first order adsorption parameters for the adsorption of As(V) on Cell-N-Cu adsorbent at 25 °C.

q_e (exp) (mmol/g)	Pseudo-first order			Pseudo-second order		
	k_1 (min^{-1})	q_e (calc) (mmol/g)	R^2	k_2 (g/mmol min)	q_e (calc) (mmol/g)	R^2
0.83	0.44	0.57	0.9711	1.4	0.88	0.9974

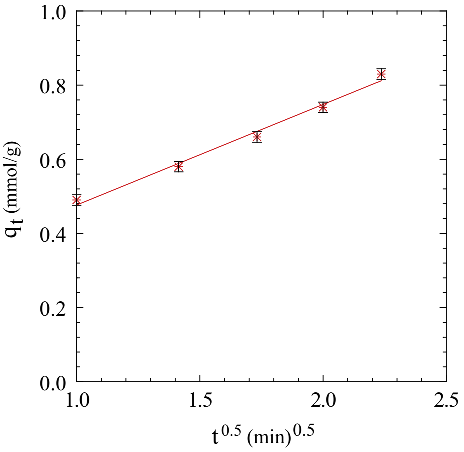


Figure 7 The intraparticle diffusion plots for the adsorption of As(V) on Cell-N-Cu adsorbent.

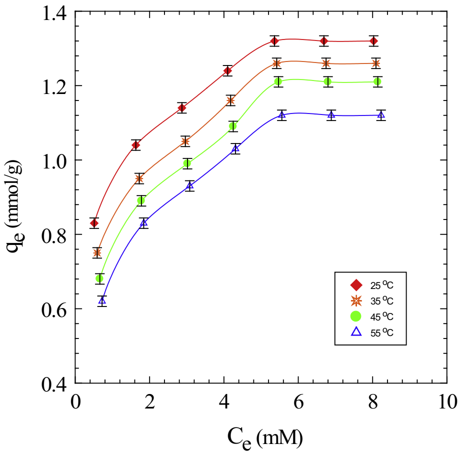


Figure 8 Adsorption isotherms of As(V) on Cell-N-Cu adsorbent at different temperatures.

processes were found to fit well the pseudo-second order more than pseudo-first order model as shown in Fig. 5 and 6 and Table 2. It is obvious that the correlation coefficients, R^2, from the second-order sorption kinetics were higher than those from the first-order kinetics. In addition, the calculated equilibrium uptake capacities from the second-order kinetics fitted well with the experimental data.

Most of the adsorption reactions take place through a multi-step mechanism comprising; (i) external film diffusion, (ii) intraparticle diffusion and (iii) interaction between adsorbate and active sites.

Table 3 Langmuir parameters for adsorption of As(V) on Cell-N-Cu adsorbent at different temperatures.

Temp. (°C)	$q_e^{(\text{exp.})}$ (mmol/g)	$Q_{\max}$ (mmol/g)	K_L (L/mmol)
25	1.32	1.41	1.98
35	1.26	1.37	1.46
45	1.21	1.34	1.19
55	1.12	1.25	1.16

To examine the rate determining step of the adsorption reaction, the uptake/time data obtained were treated according to the Fickian diffusion low (Guibal et al., 1998).

$$q_t = x + k_i t^{0.5} \tag{6}$$

where q_t was the amounts of As(V) adsorbed at time t, k_i (mmol/g min$^{0.5}$) is the intraparticle diffusion rate constant and x is an indication for the thickness of the boundary layer. The straight line obtained and the positive values of x (Fig. 7) indicate that the adsorption process using Cell-N-Cu adsorbent is controlled by the intraparticle diffusion as well as the boundary layer thickness.

3.2.3. Adsorption isotherms

The adsorption isotherms of As(V) at different temperatures were shown in Fig. 8. Obviously, At 25 °C, increasing arsenic concentrations involves an increase in the uptake As(V) till reaching the maximum uptake value of 1.32 mmol/g. Moreover, the adsorption capacity showed a decrease with increasing temperature, this may be related to the decrease of the electrostatic attraction between As(V) and Cu(II) active sites at higher temperatures.

The adsorption data were treated according to linear Langmuir equation (Gupta and Babu, 2009).

$$\frac{C_e}{q_e} = \frac{C_e}{Q_{\max}} + \frac{1}{K_L Q_{\max}} \tag{7}$$

where C_e is the equilibrium concentration of As(V) in solutions (mmol/L), q_e is the amount adsorbed at C_e (mmol/g), $Q_{\max}$ is the maximum adsorption capacity (mmol/g), and K_L is the binding constant of Langmuir which is related to the energy of adsorption (L/mmol). Plotting C_e/q_e against C_e gives a straight line with slope and intercept equal to $1/Q_{\max}$ and $1/K_L\,Q_{\max}$, respectively. The values of K_L and $Q_{\max}$ at different temperatures were reported in Table 3. The values of $Q_{\max}$ obtained at different temperatures are in good agreement with the experimental ones. This indicates that the adsorption is a monolayer and proceeds according to Langmuir model.

Degree of suitability of Cell-N-Cu adsorbent towards As(V) was estimated from the values of the separation factor (R_L) which is defined by the following equation (Deniz and Saygideger, 2010).

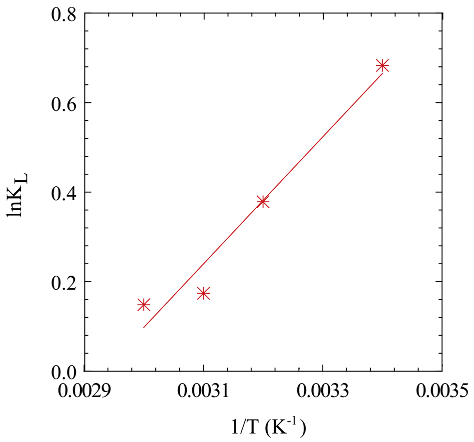


Figure 9 Van't Hoff plots for the adsorption of As(V) by Cell-N-Cu adsorbent.

$$R_L = \frac{1}{1 + K_L C_o} \tag{8}$$

where C_o is the initial concentration of the metal ion (mmol/L). The values of R_L indicates the nature of the adsorption to be irreversible ($R_L = 0$), favorable ($0 < R_L < 1$) and unfavorable ($R_L = 1$). At 25 °C, the values of R_L obtained according to the above equation were 0.274 and 0.045 for Cell-N-Cu adsorbent using initial As(V) concentrations of 1×10^{-3} M and 1×10^{-2} M, respectively. This implies that the adsorption of As(V) on Cell-N-Cu adsorbent from aqueous solutions is favorable under the conditions used in this study.

Thermodynamic parameters of the adsorption reaction were calculated using van't Hoff equation (Al-Degs et al., 2008).

$$\ln K_L = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{9}$$

where K_L is Langmuir binding constant (L/mmol), R is the universal gas constant (8.314 J/mol K), T is the absolute temperature (Kelvin). Plotting of $\ln K_L$ against $1/T$ gives straight lines with slope and intercepts equal to $-\Delta H^\circ/R$ and $\Delta S^\circ/R$, respectively (Fig. 9). Negative values of both ΔH° and ΔS° for Cell-N-Cu adsorbent (Table 4) indicate an exothermic adsorption reaction accompanied with more ordered state in activated complex due to strong interaction between $HAsO_4^{2-}$ and surface active sites of the adsorbent (Gode and Pehlivan, 2003). The Gibbs free energy (ΔG°) values of the adsorption reactions were also obtained using the following relation and given in Table 4.

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{10}$$

The negative values of ΔG° indicate that the adsorption is spontaneous. For Cell-N-Cu adsorbent, ΔG° values decrease as temperature increase which indicates that the process of adsorption of As(V) become less favorable at higher temperatures. This may be attributed to the exothermic nature of the adsorption process. The data given in Table 4 for Cell-N-Cu adsorbent show that $|T\Delta S^\circ| < |\Delta H^\circ|$ at all temperatures. This indicates that the adsorption reaction is dominated by enthalpic rather than entropic changes. These thermodynamic features of the adsorbent towards the adsorption of As(V) confirm our suggestion on the effective role of the structural characteristics of the adsorbent on the strength of interaction.

Table 4 Thermodynamic parameters for adsorption of As(V) on Cell-N-Cu adsorbent.

Temp.(K)	ΔH° (kJ/mol)	ΔS° (kJ/mol K)	ΔG° (kJ/mol)	$T\Delta S^\circ$ (kJ/mol)
298	−14.8	−0.045	−1.5	−13.28
308			−1.1	−13.73
318			−0.7	−14.17
328			−0.2	−14.62

Table 5 ICP results of wastewater real sample before and after treatment with the investigated adsorbent.

Ion	C_i (mg/L)	C_f (mg/L)	Removal ratio (%)
SO_4^{2-}	110.6	90.6	18.08
CO_3^{2-}	100.2	86.3	13.87
PO_4^{3-}	3.00	2.10	30.0
NO_3^-	1.26	1.18	6.35
Cl^-	90.4	80.2	11.28
As(V)	102.2	28.4	72.21

3.3. Regeneration experiments

Sorption/desorption cycles were performed repeatedly using ammonium hydroxide as an eluent. The efficiency of regeneration was found to be 98%. This value was reported after three cycles with standard deviation of ±1%. This finding reveals the stability of the adsorbent for the repeated use.

3.4. Selective adsorption of As(V) from a real wastewater sample

A real sample was obtained from a wastewater treatment plant containing sulfate, chloride, carbonate, nitrate and phosphate ions, Their concentrations were measured before and after treatment with the adsorbent and the data were recorded in Table 5 which are considered as competing ions in the arsenic adsorption process. It was expected that sulfate and carbonate ions would noticeably decrease the adsorbent efficiency due to their divalent state, but the obtained results indicate no significant influence on the capacity of studied adsorbent for arsenic adsorption. These results make Cell-N-Cu promising in arsenic selective separation in wastewater treatment.

4. Conclusion

Copper loaded (Cell-N-Cu) adsorbent was prepared and investigated. The adsorbent obtained is characterized by a fast and high adsorption capacities towards As(V) from aqueous media at natural pH (8.4). The equilibrium adsorption was reached within 5 min and reached 1.32 mmol/g. The adsorption process was found to follow pseudo-second order kinetics. The regeneration efficiency of the loaded adsorbent was found to be 98% ± 1% for As(V) using ammonium hydroxide. High arsenic uptake capability obtained from real sample results and cost-effectiveness of modified cellulose sorbent made it potentially attractive for arsenic removal from different waste streams.

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