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Quaternized hydroxyethyl cellulose ethoxylate and membrane separation techniques for arsenic removal

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ABSTRACT

Quaternized hydroxyethyl cellulose ethoxylate (QHECE) was combined with a regenerated cellulose membrane (10 kDa), and its potential for arsenic removal was investigated. The arsenic removal was evaluated by polymer-enhanced ultrafiltration (PEUF) using QHECE as a sorbent. The parameters studied during PEUF were pH, optimum concentration of polymer, effect of interfering ions and maximum retention capacity. Additionally, the electrochemical oxidation of As(III) to As(V) in the presence of QHECE was performed to remove total arsenic from solution. The PEUF results showed a retention of 63% for As(V) and null retention for As(III) at pH 9. This retention capacity was obtained using 100 mg of QHECE and 30 mg/L arsenic. Moreover, the As(V) retention did not decrease in the presence of interfering ions. The maximum retention capacity was 56 mg As/g polymer. The electrochemical oxidation of As(III) to As(V) was successfully performed using QHECE as the supporting electrolyte. After oxidation, the retention of arsenic by PEUF reached 90%. These results are promising and indicate new potential applications for these types of cationic polysaccharides.

Keywords: Arsenic; Electrolyte; Membrane; Polysaccharide; Remediation

1. Introduction

Cellulose is an abundant polysaccharide, and it is the most abundant renewable resource worldwide [1,2]. The advantages of cellulosic materials are that they are safe for living organisms, reproducible, recyclable and biodegradable. Many technologies have been used to produce cellulosic materials that are modified for many applications and industrial uses [3]. Chemical modification or functionalization of natural polymers seeks to improve some properties, such as affinity and selectivity, towards ions for environmental applications [4–6]. Hydroxyethyl cellulose has recently been modified with quaternary ammonium monomers, resulting in a cationic polymer based on cellulose with optimum viscosity [7]. This is a highly modified polymer used in the manufacturing of hair care products because of its exceptional power to restore damaged hair and improve the wettability, appearance and feel of dry hair. In recent years, scientists have shown interest in studying the solution properties and adsorption behaviour of these cellulosic polymers [8].

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On the other hand, heavy metals and metalloids are the most dangerous water contaminants, and because their concentration is continuously increasing, they have also attracted attention from researchers around the world [9,10]. Arsenic is one of the most common pollutants found in natural and waste waters [11]. This metalloid can be highly toxic even at low concentrations, and the World Health Organization has established a maximum permissible limit for arsenic of 10 μg/L [12]. In nature, it is possible to find arsenic at different concentrations, which generates serious environmental problems in several countries. Arsenic is present in aqueous systems primarily as As(V), arsenate, and As(III), arsenite. As(III) is more toxic than As(V); however, As(V) is more abundant, particularly in superficial waters [13].

There are currently several technologies for removing arsenic that employ membranes or polymeric materials, including reverse osmosis, nanoprocesses, micro and ultrafiltration, ion-exchange resins and water-soluble polymers [14]. It has been determined that the combination of ultrafiltration and water-soluble polymers, also called polymer-enhanced ultrafiltration (PEUF), is an efficient technique for removing ions as long as there is a suitable metal–polymer interaction. In ultrafiltration systems, the membrane functions as a molecular size barrier; however, the pore size is too large to retain metal ions without the ions being in colloidal form [15]. The formation of polymer–metal species with high molecular weights leads to the retention of metal ions [11,15,16]. The greatest advantage of the PEUF method is that it is performed in homogeneous media, thus largely avoiding the phenomenon of mass transfer or diffusion that occurs in heterogeneous media [12,15]. PEUF has been used with synthetic polycations to remove As(V) species from water. The removal of As(V) was efficient because arsenate is a negatively charged species over a wide range of pH [11]. Regarding trivalent arsenic, we have previously reported the efficient removal of As(III) via electrochemical oxidation and subsequent PEUF using synthetic polymers [17].

This study is related to the removal of arsenic by PEUF using water-soluble quaternized hydroxyethyl cellulose ethoxylate (QHECE) as both an extracting agent and a supporting polyelectrolyte. The parameters studied during PEUF were pH, optimum concentration of polymer, effect of interfering ions and maximum retention capacity of the polymer. After optimization of PEUF, the electrochemical oxidation of As(III) to As(V) was performed in the presence of QHECE and subsequently coupled to PEUF to remove total arsenic from aqueous solutions.

2. Experimental

2.1. Ultrafiltration procedure

In this study, the water-soluble polymer was a commercial QHECE purchased from Aldrich. Two polymer fractions were separated using ultrafiltration membranes with molecular weight cut-offs (MWCOs) of 30 and 100 kDa; then, the polymer fractions were isolated by freeze-drying. The fraction used in this study was greater than 100 kDa.

The PEUF system includes an ultrafiltration cell (Amicon 8050) with a magnetic stirrer containing a regenerated cellulose membrane with a MWCO of 10 kDa (Ultracel PLGC, Millipore), a reservoir (Amicon RC800), a selector, and a nitrogen gas cylinder as the pressure source. This system is described in the literature [15].

In this study, two different PEUF modes were used to remove arsenic. The first mode was the washing method. In this method a polymer solution is contacted with a arsenic solution in the inside of filtration cell at a given pH and ionic strength. System is put into operation, and as result of applied pressure, a reservoir-contained solution stream enter to the cell and equal volume of solution pass through membrane to the permeate stream. In this process, the solution in the cell is continuously washed [15]. In this experiment, QHECE is dissolved in deionized water and another solution coming from reservoir at a certain pH is added to it. Subsequently, washing was performed with water adjusted to a constant pH of 3, 6 or 9, which was adjusted by adding 1×10^{-1} mol/L HNO_3 or NaOH (Merck), respectively. The pH value was measured using a model 330 pH meter from inoLab WTW. The resulting polymer–arsenic mixture was stirred for 1 h at room temperature. Then, the solution was introduced to the ultrafiltration cell and washed with reservoir water at the same pH (see Fig. 1). All ultrafiltration studies were performed under a total pressure of 3.5 bar at room temperature using an ultrafiltration membrane of regenerated cellulose with a MWCO of 10 kDa. The total volume (20 mL) in the cell was held constant, and measurements of 20 mL were collected by filtration up to a total volume of 100 mL.

Using the washing method, two quantities were defined. In Eq. (1), retention (R) denotes the fraction of arsenic remaining in the cell, and in Eq. (2), the filtration factor (Z) denotes the volume exchange ratio in the reservoir.

$$R = [\mathrm{As}]_{\mathrm{cell}}/[\mathrm{As}]_{\mathrm{i}} \tag{1}$$

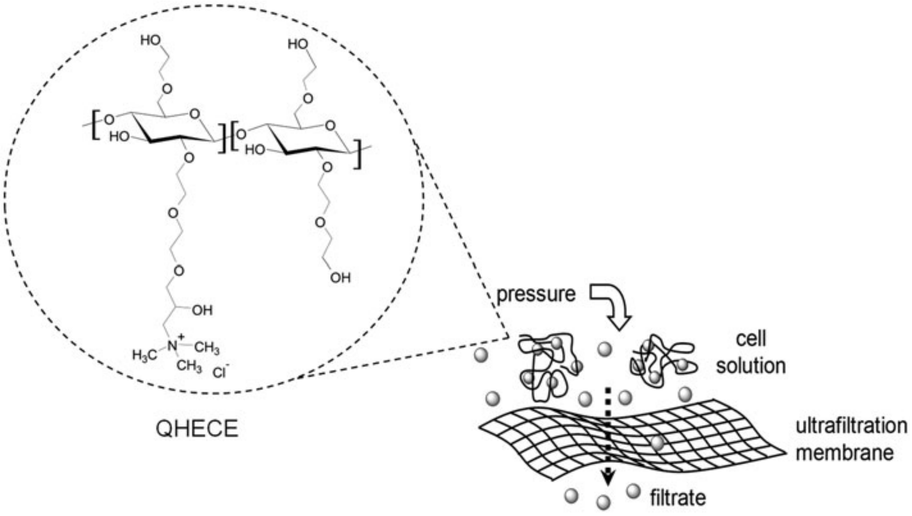

Fig. 1. Principle of polymer-enhanced ultrafiltration and chemical structure of QHECE.

where $[As]_{cell}$ is the amount of arsenic that is retained by the polymer in the cell and $[As]_i$ is the initial amount of arsenic in the feed. The filtration factor (Z) is the ratio between the total permeate volume (V_p) and the retentate volume (V_r):

$$Z = V_p/V_r \tag{2}$$

Using the experimental data, we can then draw a graph (retention profile) in which % R is represented as a function of Z. The washing method was also performed in a blank experiment (in the absence of the QHECE), which was included to evaluate the interaction of the regenerated cellulose membrane with arsenic.

The second mode presented in this work is the enrichment method, which determines the maximum retention capacity (C) of the QHECE. The arsenic-containing solution passes from the reservoir through the ultrafiltration cell filled with the QHECE solution. Both the cell and reservoir solutions are adjusted to the same values of pH. In this study, both the reservoir solution and the cell solution contained 35 mg/L arsenic, while the cell solution also contained 100 mg of QHECE. The total permeate volume of the LPR system was set to 160 mL. The maximum retention capacity of the QHECE was determined using the enrichment method, and it is defined as follows (see Eq. (3)):

$$C = (MV)/Pm \tag{3}$$

where Pm is the amount of polymer (g), M is the initial concentration of arsenic (mg/L), and V is the volume of permeate (mL) that passes through the membrane. The maximum arsenic retention capacity was calculated when 160 mL of permeate was collected.

The concentration of arsenic was measured by atomic absorption spectroscopy, AAS, using a Perkin–Elmer (Waltham, MA, USA) 3100 spectrometer, and the quantity retained was determined from the difference from the initial concentration. The calibration curve for the arsenic measurements was constructed between 5 and 50 mg/L.

2.2. Electrochemical oxidation

Electrochemical experiments were performed using a CHI 660B electrochemical potentiostat/galvanostat (CH Instruments) using a conventional three-electrode system. The voltages were referenced with respect to Ag/AgCl in a 3 mol/L KCl reference electrode. A platinum wire or a platinum basket was used as the counter electrode in the same electrochemical cell. Gold disc electrodes (3-mm diameter) were polished with 1-mm diamond for the analytical experiments. Cyclic voltammograms were recorded after three cycles when signal stabilization was achieved. Subsequently, the voltammograms remained constant for 10 continuous cycles.

Extensive oxidations of As(III) (from $NaAsO_2$, Merck, reagent grade) to As(V) species were performed in air at room temperature and under constant stirring at 1,000 rpm on a large area of gold electrodes

($1\ \mathrm{cm}^2$). In a typical experiment, the electrolysis cell was charged with 20 mL of solutions containing a QHECE used as the supporting electrolyte and As(III) at different concentrations. The voltage applied to achieve As(III) oxidation on gold was in the range of 0.6–0.8 V. The electrolysis was conducted in a one-compartment cell, without a separator between the working electrode and the auxiliary electrode (a platinum basket). During the electrolysis, the solution was stirred at 1,000 rpm.

3. Results and discussion

3.1. *Effect of pH on the retention of As(V)*

The effect of pH on the removal of As(V) was investigated. The experiment was performed using a regenerated cellulose membrane with a MWCO of 10 kDa, and this pore size in molecular weight is considerably lower than that of the QHECE used. During this study, QHECE was investigated as an anion exchanger. The retention results as a function of pH are presented in Fig. 2.

First, the blank experiment (without QHECE) revealed that the cellulose membrane does not remove As(V) when the experiment reaches $Z = 5$. This result means that the membrane does not have strong interactions with arsenate species and that these species pass rapidly to the filtrate. The results regarding As (V) retention in the presence of QHECE at different pHs showed that As(V) is more easily retained at pH 9 than at pH 6 or 3. The retention of As(V) at $Z = 5$ by QHECE was approximately 46, 53 and 63% at pH 3, 6 and 9, respectively. These results are in a good agreement with the data reported in the literature [15]. This behaviour can be attributed to the presence of different arsenate species in aqueous media depending on the pH. At pH 3, the monovalent anionic species ($\mathrm{H_2AsO_4^-}$) is in equilibrium with the coupled salt. At pH 6, the monovalent ($\mathrm{H_2AsO_4^-}$) and divalent ($\mathrm{HAsO_4^{2-}}$) species exist in the equilibrium [18]. The polymer's interaction capacity is primarily due to the presence of a quaternary ammonium group in QHECE. It was observed that the divalent species have a higher affinity towards the QHECE than that of monovalent species. It is well known that the interactions between ion exchange groups of the resins and ions in solution increase with the ionic charge. These interactions are produced through the anion exchange between the chloride counterion of QHECE and the arsenate anion. Moreover, the shape of the retention curves at pH 3, 6 and 9 are constant from $Z = 3$ to $Z = 5$. This result is important because it means that the interaction is sufficiently strong and that arsenic does not pass through the membrane when the polymer-As solution is washed with water at a certain pH.

3.2. *Study of the amount of polymer on the retention of As(V)*

Using the washing method, the removal of As(V) was optimized by varying the amount of polymer at pH 9. In the ultrafiltration cell, the amount of polymer was varied (25, 75 and 100 mg) while maintaining a constant concentration of arsenate (30 mg/L). Fig. 3 shows that the retention of arsenic increased as the amount of polymer increased. The results for As(V) showed that when using 25, 75 and 100 mg, the retentions at $Z = 5$ were 53, 52 and 63%, respectively. The differences in arsenic retention as a function of the amount of polymer may be due to conformational changes of the polymer, which was dissolved at the

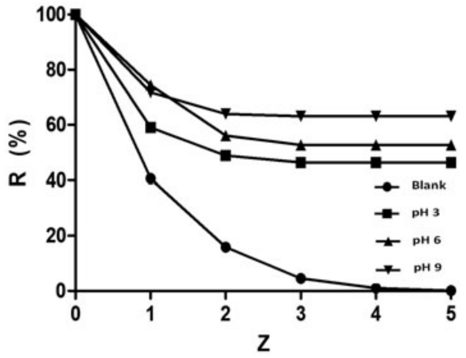

Fig. 2. Retention profiles of As(V) using 100 mg of QHECE as an extracting polymer and a regenerated cellulose membrane at different pH values with 30 mg/L As(V) in the feed and pressure of 3.5 bar.

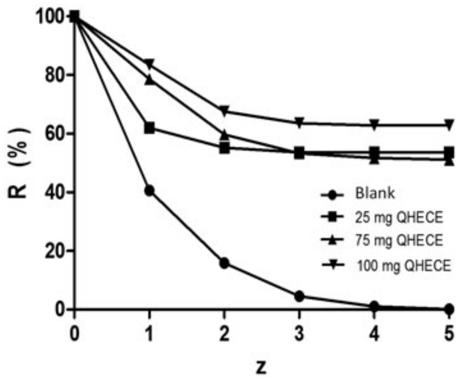

Fig. 3. Retention profile of 30 mg/L As(V) at pH 9 as a function of the amount of polymer using a regenerated cellulose membrane and pressure of 3.5 bar.

same volume (20 mL). These conformational changes influence the electrostatic interactions (attractive or repulsive) between the cationic polymer and the anion to be removed from solution [15].

3.3. Effect of competitive anions on the retention of As(V)

The effect of competitive anions, such as Cl^-, SO_4^{2-} and PO_4^{3-}, on the removal of As(V) was investigated. The experiments were conducted separately at pH 9 using 100 mg of QHECE, 30 mg/L As(V), pressure of 3.5 bar and a regenerated cellulose membrane. The results indicate that when chloride and sulphate were added to the solution, the As(V) retention did not decrease (see Fig. 4). In contrast, when 30 mg/L Cl^- or SO_4^{2-} was added to the polymer–As solution, the retention of As(V) increased to 92 and 74%, respectively. This result can likely be explained by the increase in the ionic strength of the solution, which could increase the amount of functional groups available for ion exchange [19].

However, the effect of PO_4^{3-} ions on As(V) removal was different. In this case, the retention of arsenate decreased compared with the experiment in the absence of phosphate. According to the literature, the order of interference in the retention of arsenic is as follows: trivalent ions > divalent ions > monovalent ions. The effect of added salts on arsenic binding to the polymer can be understood as being due to the competition between arsenate and other anions for binding sites on the polymer. The affinity of anions towards binding onto the polymer is similar to the behaviour observed in the ion-exchange resin containing ammonium groups when removing arsenic through the ion-exchange process [19]. These results demonstrate the strong selectivity for removing As(V) from aqueous solution.

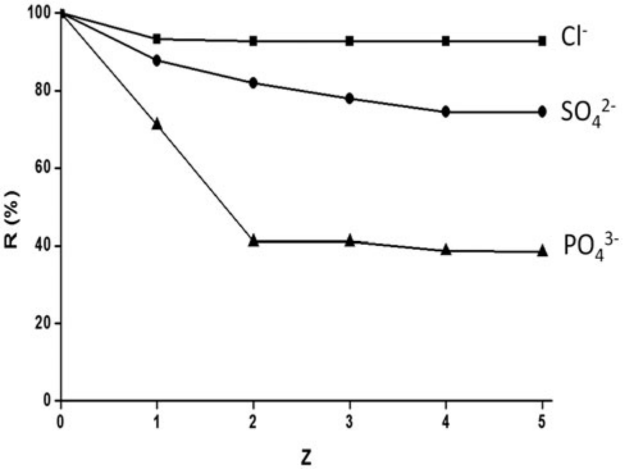

Fig. 4. Retention profiles of 30 mg/L arsenate using 100 mg of QHECE and a regenerated cellulose membrane at pH 9 and a pressure of 3.5 bar in the presence of 30 mg/L Cl^-, SO_4^{2-} and PO_4^{3-}.

3.4. Sorption–desorption process

To study the arsenic sorption–desorption process, the enrichment method and the washing method were used. The first step of the experiment consisted of saturating QHECE through the enrichment method using a 35 mg/L As(V) solution at pH 9 and 100 mg of QHECE collecting a total filtrate volume of 160 mL. The concentration of As(V) was measured in the filtrate.

The blank test was the experiment without QHECE in the cell. The results of the blank experiment revealed higher concentrations of As(V) in the first tubes (see Fig. 5(A)). During the enrichment of QHECE, the same experiment was performed; however, in this case, QHECE was incorporated into the cell. With the results of the analytical measurements, the profile of As(V) in the filtrate vs. the volume of filtrate was obtained. When the QHECE curve reached a plateau (i.e. was equivalent to the blank) under our experimental conditions, we calculated the maximum retention capacity. After reaching the saturation of QHECE, the polymer–As(V) solution retained in the cell was washed with water buffered at pH 2 from the reservoir. The desorption process was applied to determine the capacity of As(V) delivery.

The enrichment process (sorption) reached the maximum retention capacity at pH 9. The value of the maximum retention capacity of the first sorption was 56 mg As/g QHECE. After the sorption process, desorption was performed by changing the pH from basic to acidic using HCl or HNO_3. Fig. 5(B) shows the process of As(V) desorbing from the polymer when the polymer-As(V) is in contact with the acidic solution at pH 2. The polymer released between 60 and 70 mg/L of arsenic in the first tube of filtrate, and then, the concentration decreased until reaching 100 mL of filtrate. These results showed that it is possible to release the arsenic retained during the enrichment method by washing with an acidic solution. More cycles must to be performed to determine the regeneration of the polymer.

3.5. Electrochemical oxidation of As(III) to As(V)

Several studies have shown that electrochemical techniques are used to oxidize arsenic with various

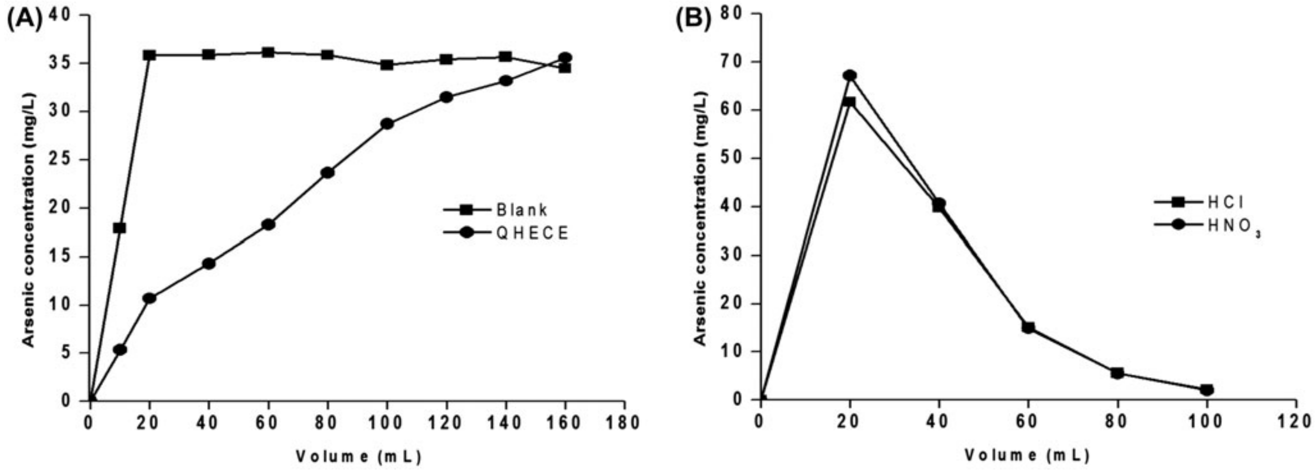

Fig. 5. Sorption–desorption process of As(V) using QHECE combining (A) enrichment at pH 9 and (B) the washing method at pH 2.

electrode materials, such as platinum and gold electrodes. In our previous work, we studied the electrochemical oxidation of As(III) to As(V) in the presence of synthetic water-soluble polymers containing quaternary ammonium groups and different metallic and modified electrodes [15,17]. In this work, for the first time, we use a modified polysaccharide (QHECE) as a supporting electrolyte, and we analyse its properties in the electrochemical oxidation of As(III) to As(V) using gold electrodes.

Fig. 6 presents the typical cyclic voltammetry response (scan rate of 10 mV/s) recorded at a gold electrode when adding increasing amounts of As(III) to a solution containing 100 mg of QHECE. The peak height obtained at approximately +0.75 V increased linearly and showed a good response with the addition of the arsenite species. The linear range extends from 5 to 30 mg/L. The stability of the gold electrodes was investigated, but only to a limited extent. A marginal loss of activity towards arsenic oxidation

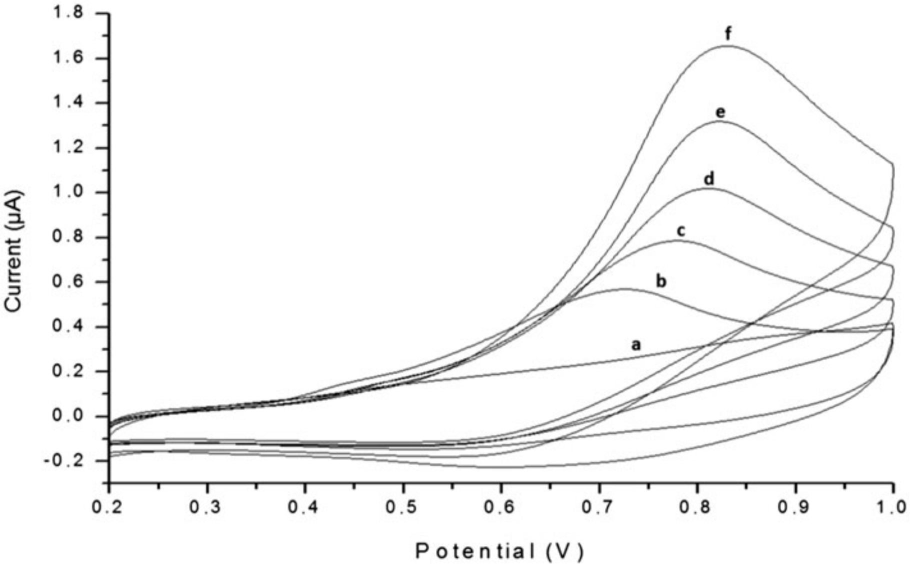

Fig. 6. Cyclic voltammetry in the presence of increasing amounts of As(III) recorded on a gold electrode and with 100 mg of QHECE as a supporting electrolyte. Curve *a* is the blank in the absence of As(III), and curves *b*, *c*, *d*, *e* and *f* are in the presence of 5, 10, 15, 20 and 30 mg/L As(III), respectively; the scan rate was 10 mV/s between 0 and 1.0 V vs. Ag/AgCl.

(less than 10%) after a series of at least 10 experiments was observed. These results reveal that the use of QHECE as a supporting electrolyte in the oxidation process is an advantage for developing new systems for arsenic monitoring and removal. In the next step, this calibration curve was used to measure the remaining concentration of As(III) during the advancement of the extensive oxidation process.

3.6. Extensive electrochemical oxidation of As(III) to As(V)

The extensive electrochemical oxidation of As(III) to As(V) using gold macroelectrodes was tested in solutions containing a QHECE as the supporting electrolyte. The experiment was conducted with a solution of arsenite (30 mg/L) and 100 mg of QHECE in 20 mL of solution.

To obtain the complete conversion of As(III) to As (V), the QHECE–arsenite solution was electrolysed at +0.8 V vs. Ag/AgCl. The progress of the oxidation of As(III) to As(V) was monitored *in situ* using cyclic voltammetry with a gold analytical electrode.

The results showed good behaviour for both extensive oxidation and for analytical measurements. The use of QHECE as a support in the electrochemical process showed a decreasing current response (see Fig. 7). The results showed the conversion of As(III) to As(V), reaching the theoretical charge calculated for the process under these conditions. The theoretical charge for the complete oxidation of arsenite solution (30 mg/L) was 1.54 coulombs, as calculated according to Coulomb's Law (Q = number of electrons × 96,500 × As(III) concentration). The conversion of As(III) to As(V) was estimated at each point following the decrease of voltammetric peaks. The decrease of the peak current of As(III) followed an linear slope from a charge of 0.3 to 1.2 C; then, the peak currents decreased rapidly to reach the complete As (III) oxidation. The next step is to study the removal of this electro-oxidized arsenic solution through the PEUF washing method.

3.7. Removal of the electro-oxidized arsenic

We previously determined the influence of pH on arsenate retention by QHECE combined with ultrafiltration membranes. The interactions between QHECE and arsenate species were effective under basic pH due to the speciation of arsenic. For this reason, after the extensive oxidation of As(III) to As(V), the pH of the electrolysed solution was adjusted to 9. Next, the resulting solution was assayed with the PEUF using the washing method. The removal experiments were comparatively studied using As(III) (not electrolysed) and As(V) (previously oxidized).

Fig. 8 shows the retention profile of arsenic (30 mg/L) by QHECE. In this profile, it is possible to observe that the retention of As(III) without oxidation is null at $Z = 3$. This result can be explained by the speciation of arsenite in aqueous solution, which is not negatively charged at pH 9 [15]. However, the retention is improved through the use of a previously oxidized arsenite solution, reaching 90% at $Z = 5$. This retention curve is constant from $Z = 1$ to $Z = 5$. The interaction between QHECE and the previously oxidized arsenic species is strong. It is due to electrostatic

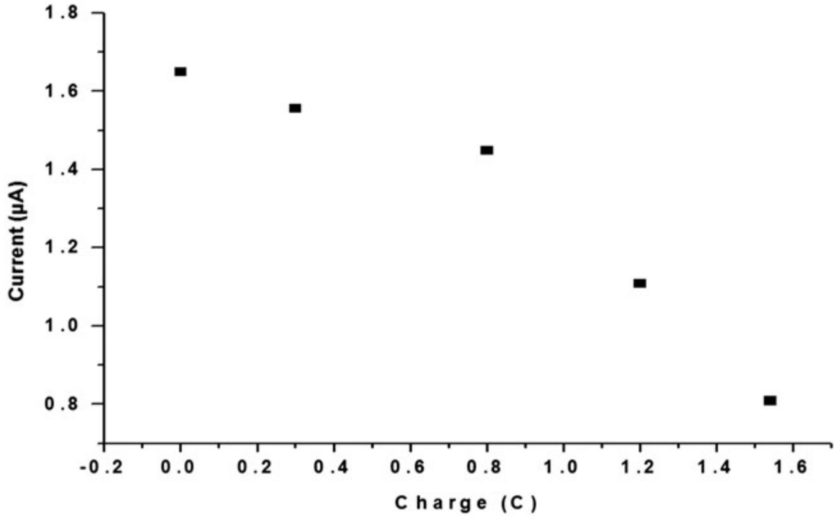

Fig. 7. Plot of the As(III) oxidation current recorded at the gold microelectrode vs. charge consumed during electrolysis.

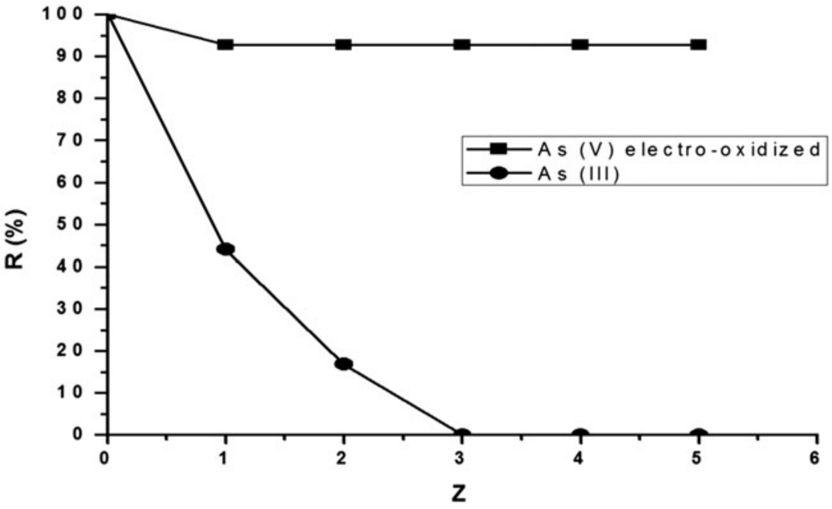

Fig. 8. Retention profile of electro-oxidized arsenic (black circle is without oxidation, and black square is previously oxidized) by a QHECE at pH 9.

Table 1
Comparison of arsenic removal capacities of different water-soluble polymers

Polymer	*R* (%)	Refs.
P(ClAETA)	60	[17]
P(GMA-NMG)	60	[17]
PDDA	100	[20]
QHECE	90	–

interaction between positively charged polymer and negatively charged arsenate. The arsenic species are not passing through the membrane and therefore are removed from the aqueous solution.

Table 1 shows the comparison of arsenic removal capacity of different water-soluble polymers. In all the cases, arsenite was oxidized to arsenate in presence of the water-soluble polymer before the ultrafiltration process. Poly 2-(acryloyloxy)ethyl trimethylammonium chloride, P(ClAETA) shows 60% of arsenic removal. Poly(glycidyl methacrylate N-methyl-D-glucamine), P (GMA-NMG) also shows 60% of arsenic removal. Poly (diallyldimethyl ammonium) chloride, PDDA, showed almost 100% of arsenic removal, but in this case the oxidation was performed using an efficient polymer-metal nanostructured anode [20]. In the case of QHECE reported in the current study, the maximum removal of arsenic was 90%.

4. Conclusions

In this study, QHECE was combined with a regenerated cellulose membrane, and its potential for removing arsenic was systematically analysed by PEUF. The parameters studied were pH, optimum concentration of polymer and the effect of competitive anions. The results showed a retention of 63% for As (V) and null retention for As(III) under optimized conditions (pH 9 and 100 mg of polymer). The As(V) retention does not decrease in the presence of interfering ions such as chloride and sulphate. The maximum retention capacity of QHECE was 56 mg As/g polymer.

Finally, the electrochemical oxidation of As(III) to As(V) was successfully performed using QHECE as a supporting electrolyte during both electroanalysis and extensive oxidation. After oxidation, the retention of arsenic by PEUF reached 90%.

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