



New material for arsenic (V) removal based on chitosan supported onto modified polypropylene membrane

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Abstract

This paper presents a new material easily synthesized and with low cost, with the possibility of remove arsenic and the potential capability for the remediate water bodies. In this work, the efficiency in removing arsenic of the chitosan, supported onto modified polypropylene membrane, was studied using an aqueous As(V) solution of 0.4 mg/L, achieving a removal efficiency of 75%, which corresponds to an adsorption capacity of 0.031 mg/g. The As(V) adsorption depends on pH and the degree of chitosan grafting on the polypropylene membrane. A pseudo-second-order equation describes the adsorption of the membrane, classifying it as a chemisorption process. The chitosan supported on the membrane was characterized by the analysis of wettability, FT-IR-ATR, SEM-EDS, XRD powder, and surface charge. The As ions coordinate to the chitosan polar groups, allowing their removal from the aqueous solution.

Keywords Arsenic(V) · Chitosan · Support · Modified polypropylene · Kinetics

Introduction

Arsenic (As) occurs in the environment from natural and anthropogenic sources. Anthropogenic influence has been increasing in recent years, causing environmental and health problems worldwide (Wang et al. 2019). The toxicity of arsenic is related to the chemical and oxidation state, so the trivalent form is more toxic than pentavalent arsenic (Hughes 2002). The health problems of arsenic for humans and other living organisms have been recognized as a significant problem. The International Agency for Research on Cancer (IARC) classified arsenic and its compounds as carcinogens (Teixeira et al. 2020). The effects of arsenic on human health for the long-term exposure to contaminated water, even in low concentrations (0.01–0.05 mg L⁻¹), can cause affection to all organs of the body, with effects ranging from skin lesions,

cancer, diabetes, and lung diseases (Naujokas et al. 2013). Millions of people are exposed to arsenic through drinking water in the USA, China, México, Argentina, India, and Northern Chile. Ten countries in South and Southeast Asia (Bangladesh, Cambodia, China, India, Laos, Myanmar, Nepal, Taiwan, and Vietnam) present the highest number of people who are exposed to contaminated water with arsenic, around 110 million people (Argos et al. 2010; Singh et al. 2015).

Several new materials have been developed for the removal of pollutants from the environment. One of them is the modified polypropylene (PP) with biopolymers, providing new properties different from those of the original material, such as selectivity and chemical resistance (Hernández et al. 2016). Biopolymers became popular due to their high biodegradability and their applications as renewable catalytic substrates (Kanmani et al. 2017). In water treatment containing heavy metals, biopolymers gain attention as simple, effective, and inexpensive materials (Kwok et al. 2014).

Some biopolymers have good results for arsenic removal in recent years. For example, chitosan has been used in multiple studies for the adsorption of As(V) and As(III) (Miller and Zimmerman 2010; Bhowmick et al. 2014; Elwakeel and Guibal 2015; Wang et al. 2016; Malwal and Gopinath 2017; Kwok et al. 2018; Sherlala et al. 2019; Zeng et al. 2020).

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Chitosan effectively absorbs arsenic ions due to the presence of amines and hydroxyl groups that coordinate and electrostatic interact (Kwok et al. 2014) with the metal ions. Therefore, supported chitosan on a polypropylene (PP) membrane to remove arsenic (V) can be an excellent alternative to remediate the water bodies. Besides, chitosan is an excellent candidate due to its good properties as binder, non-toxicity, biocompatibility, selectivity, and biodegradability.

Materials and methods

Materials

Microporous polypropylene membrane with a pore size of 0.45 μm , the thickness of 114 μm , and porosity of 84.6% was provided by 3M Company, and chitosan (448877 molecular weight, deacetylated chitin), acrylic acid, benzophenone (photoinitiated radical), hydrochloric acid, potassium iodide, stannous chloride, silver diethyldithiocarbamate (AgDDTC), granular zinc (20 mesh), As(V) dibasic sodium heptahydrate arsenate ($\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$), and all reagents were used without prior purification.

Preparation of modified polypropylene-chitosan membranes

1% chitosan in acetic acid, 30% acrylic acid in deionized water, 5% benzophenone in acetone were prepared. The polypropylene membrane was cut into 4×4 cm squares, and impregnated with chitosan, acrylic acid, and benzophenone. The membranes were placed between acetates and irradiated at 350 nm for 3 h. Afterwards, rinsed with acetone to eliminate the chitosan that did no graft, they are dried under vacuum to constant weight, and the grafting degree was obtained using the following formula:

$$Gd(\%) = \frac{P_F - P_I}{P_I} \times 100 \quad (1)$$

where P_F and P_I are the modified membrane and the polypropylene membrane mass, respectively.

Characterization of modified PP-Q membranes

Membrane wettability test

Polypropylene (PP), polypropylene-chitosan (PP-Q), and polypropylene-acrylic acid (PP-AA) membranes' wettability study was performed by gravimetry method at room temperature with deionized water. Each membrane was put in contact with deionized water and weight every 5 min for 30 min.

FT-IR-ATR analysis

IR spectra (Spectrum Two) coupled to ATR were acquired at room temperature, 64 scans with a window of 500–4000 cm^{-1} and 2 cm^{-1} of resolution.

SEM-EDS analysis

Microscopy images and energy dispersed spectroscopy analysis were performed on a scanning electron microscope, JEOL JSM-6510LVV; acceleration voltage from 1 to 30 kv, coupled to an X-ray detector; energy dispersion (EDS) OXFORD, with a resolution of 137 eV. For this analysis, the membranes were vacuum dried to constant weight, and carbon tape was used to improve image quality.

Point of zero charge

A total of 50 ml of deionized water were taken in 100-ml Erlenmeyer flasks, adjusting each solution pH between 1 and 11, adding 0.1 M HCl and 0.1 M NaOH. A 4×4 cm PP-Q membrane was added to each solution, under stirring at 50 rpm, room temperature, and after 48 h, pH was measured. The point of zero charge (PZC) corresponds to the point where the curve of final pH versus initial pH cuts the diagonal.

Quantification of As(V)

Quantification of As(V) by the UV-Vis spectrophotometric method used the silver diethyldithiocarbamate complex (AgDDTC). The arsenic is reduced to arsine by zinc in an acid solution. The arsine passes through an absorbent tube containing the complex to form a soluble red complex whose color is proportional to the content of arsenic in the sample.

As(V) adsorption experiment

An amount of 1g/L stock solution of $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ in deionized water was prepared. The solutions were obtained by diluting the stock solution with deionized water. HCl was used to preserve dilutions and to control pH.

The adsorption experiments were carried out in a continuous system using Erlenmeyer flasks at 25 °C. The pH effect was studied using 4×4 cm membranes with As(V) solution at 400 mg L^{-1} for 10 min, stirring at 120 rpm using an orbital shaker. The samples were maintained at 0.1 M HCl and were analyzed to determine the residual concentration of As(V) using UV-Vis spectroscopy.

As(V), the adsorption kinetics with PP-Q membranes were evaluated by shaking eight membranes of 4×4 cm with the same graft grade (70%, 80%, and 90%). In 600 ml of As(V) solution with an initial concentration of 400 mg L^{-1} for 2 h in a continuous stirring system at 120 rpm at room temperature,

the pH of As(V) solution was adjusted to 1.0 by adding 0.1 M HCl. The pH adjustment was made, taking into account the PZC analysis. The amount of As(V) adsorbed per unit weight of membranes in time was calculated from the difference between the concentration of arsenic (V) in the solution before and after the adsorption had taken effect:

$$q = \frac{v(c_i - c_f)}{m} \tag{2}$$

where *q* is the adsorption capacity (mg/g), *v* is the volume of the solution containing the sorbate (L), *c_i*–*c_f* are the initial and equilibrium concentration of As(V) in the solution; finally, *m* is the mass of the dry membrane. Quantification of As(V) was made by the indirect UV-Vis spectrophotometric method using the silver diethyldithiocarbamate complex (AgDDTC) (Thepmane et al. 2020)

Results and discussion

Modification of the PP membrane

A graft polymerization carried out the membrane modification. The polypropylene was reacted with UV radiation to generate a stable tertiary carbon-free radical, which reacts with the α,β-unsaturated group of acrylic acid, functionalizing the polypropylene. In a second step, a chitosan nucleophilic attack to the grafted acrylic acid anchors the sugar group to the membrane, changing the physical and chemical properties of the material (Hernández et al. 2016).

Previously, our group found the optimal conditions for the membrane modification, observing that the grafting degree is proportional to the reaction time, obtaining the most significant weight gain after 3 h of the photochemical reaction. The effect of chitosan concentration showed that at 2% concentration, the solution viscosity increases, making the diffusion difficult in the PP surface, decreasing the grafting degree, and reducing the number of active sites on the modified membrane.

Characterization of modified membranes

Wettability test

The wettability analysis of the modified membrane was performed using a gravimetric method at room temperature and using deionized water. The result gave information on the change of the material hydrophilicity and its capability to interact with contaminated water.

The water affinity of PP-AA increases 395%, and the PP-Q reaches 454% in less than 5 min, using PP as a reference. This test also showed us that the polypropylene was successfully

modified with polar groups that change the native polypropylene hydrophobicity (see Fig. 1).

FT-IR-ATR analysis

The FT-IR analysis also confirmed the chitosan grafting on the surface of the membrane, showing the sugar functional groups on the polypropylene membrane. The polypropylene showed the vibrations corresponding to the –CH₃ (2951 cm⁻¹, 2868 cm⁻¹, 1377 cm⁻¹) and –CH₂ (2919 cm⁻¹, 2840 cm⁻¹, 1458 cm⁻¹) groups in spectrum A of Fig. 2. The signals of the grafted acrylic acid are presented in spectrum B, as the carbonyl group vibrates at 1711 cm⁻¹. Finally, the PP-Q membrane (spectrum C) shows the vibration of the hydroxyl and amino groups, essential for the confirmation of the presence of chitosan grafted onto polypropylene. The NH₂ signal is observed at 1649 cm⁻¹, the vibration of C–O–C in 1165 cm⁻¹, observing a shift of the C=O groups at 1709 cm⁻¹.

EDS elemental analysis

The elemental analysis corroborated the polypropylene membrane modification. PP (C₃H₆) results confirm the empirical equation, which is calculated as 85.7% carbon and 14.29% hydrogen, and for PP-AA membrane (C₆H₁₀O₂) 63% carbon, 8.7% hydrogen, and 28% oxygen. A considerable amount of nitrogen was found for PP-Q membrane from the amino groups present in this biopolymer (Table 1).

SEM analysis

The scanning electron microscopy (SEM) image allowed us to observe the PP-AA and PP-Q membrane morphology through the functionalization. Fig. 3 shows the thickening of the fibers in the following order PP-Q>PP-AA>PP.

Point of zero charge

Point of zero charge (PZC) is the pH with zero net surface charge and this parameter affects the

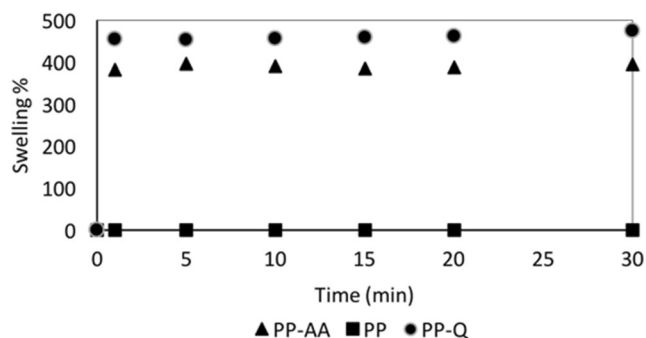


Fig. 1 Wettability process of modified membranes (PP, PP-AA, PP-Q)

Fig. 2 FT-IR-ATR spectra of (A) PP, (B) PP-AA, (C) PP-Q

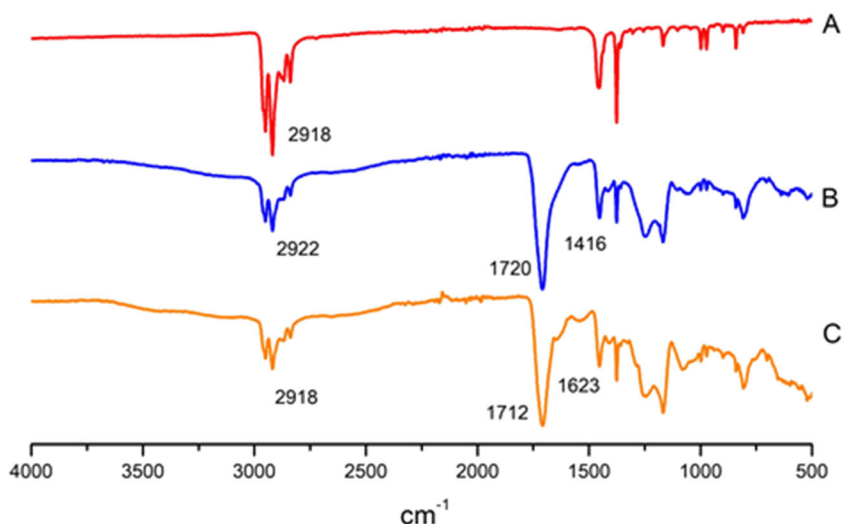


Table 1 Semiquantitative elemental analysis of PP, PP-AA, and PP-Q

Membrane	Carbon	Hydrogen	Oxygen	Nitrogen
PP	85.50	13.92	–	–
PP-AA	71.36	–	28.6	–
PP-Q	84.70	–	7.32	7.94

characteristic adsorption of PP-Q membrane. In this study, the PZC was determined by the drift method, evaluating within a pH interval from 1.0 to 11. The PZC value found was 7.01, as shown in, Fig. 4. PZC helps us to understand the effect of pH and the adsorption mechanism. The surface charge of the membrane was divided into two regions, one positive and the other negative. Below 7.01, the surface charges will be positive; therefore, the membrane surface will attract anions and repel cations. The arsenic anions interact with the positive membrane charge and remove those anions from the aqueous solution. But above 7.01, the surface charges will be negative. However, the electrostatic

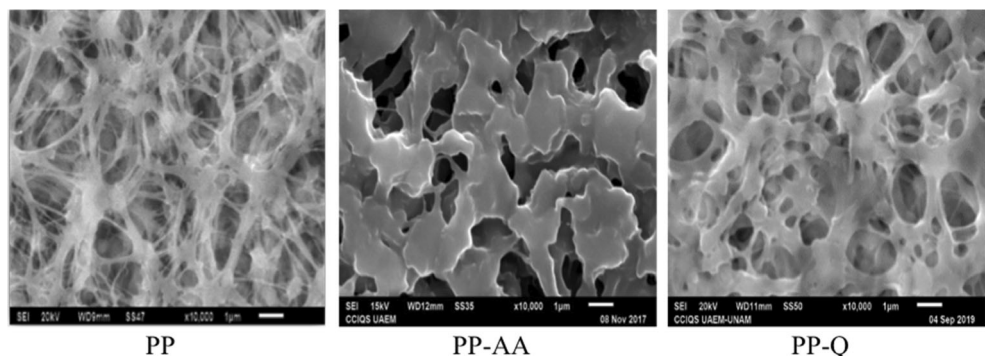
attraction between the membrane and the cations will occur, while the repulsion will happen with the anions.

Speciation of chitosan

The PZC for PP-Q was at 7.01 and decreasing below 4.5, where the amino group protonation is 99%, as shown in Fig. 5. Therefore, positively charged PP-Q can adsorb arsenic acid. As shown in Eq. (3):



Fig. 3 SEM images ×10,000 of PP membranes such as PP, PP-AA, and PP-Q



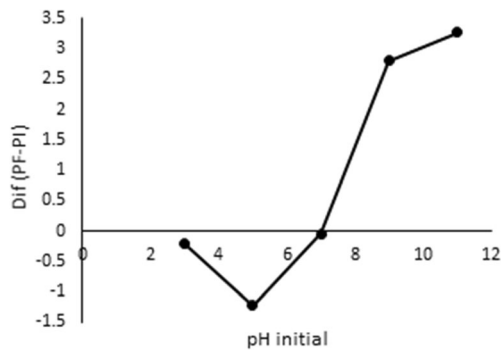


Fig. 4 PZC analysis of PP-Q membrane

Adsorption of As(V)

The effect of pH

The pH parameter affects the removal process depending on arsenic speciation and chitosan surface charge; see Fig. 6. We changed the initial solution's pH from 4.3 to 1.8. Fig. 7 showed the removal efficiency, and the adsorption capacity increased as the pH decreased from 4 to 1.8. The highest adsorption capacity of As (V) was reached around pH 1.

The effect of grafting degree

The grafting degree of chitosan on the membrane (PP-Q) is an important consideration in the adsorption process. The arsenic (V) removal efficiency was expected to increase with increasing chitosan graft on the membrane because more active sites for adsorption would be provided. However, the arsenic (V) removal was not affected by the grafting degree because the removal of the different degrees of the PP-Q membrane gave a similar performance. PP-Q probably has the same active sites available for the arsenic adsorption onto the modified membrane, see Fig. 8.

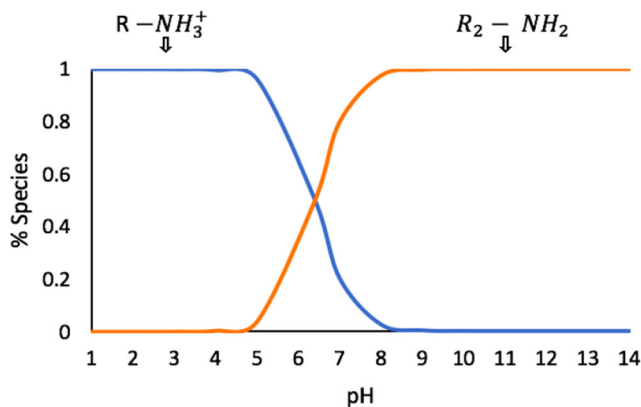


Fig. 5 Speciation diagram for PP-Q

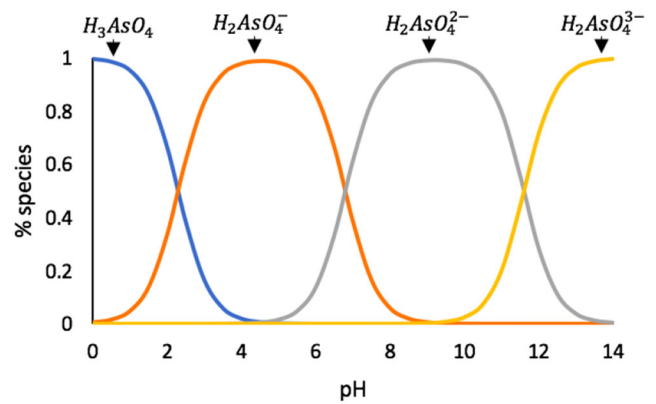


Fig. 6 As(V) speciation diagram a function of pH

Adsorption kinetics

The kinetics of As(V) adsorption using PP-Q membranes was analyzed. Three kinetic models were evaluated. One of them was the pseudo-first-order model that describes the adsorption process as physisorption. Second, the pseudo-second-order, which describes the process as chemisorption, and third, the Elovich model, that the adsorption of contaminants in an aqueous solution. The following equations represent models of the pseudo-first-order (Eq. 4), pseudo-second-order (Eq. 5), and Elovich model (Eq. 6).

$$\log \log (q_e - q_t) = \log q_e - \frac{K_L}{2.303} \times t \quad (4)$$

$$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (5)$$

$$q_t = a + 2.303 b \log \log t \quad (6)$$

where:

- K_L = Pseudo-first-order constant or sorption rate (L/min)
- q_e = Amount of solute adsorbed at equilibrium (mg/g)
- q_t = Amount of solute adsorbed at time t (mg/g)
- t = Time (min)
- K_2 = Pseudo-second-order constant (g/mg min)

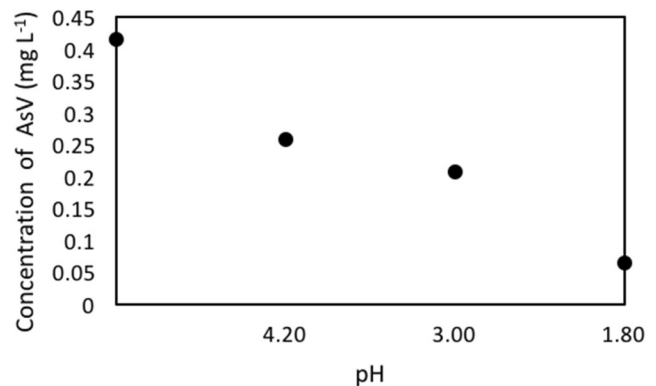


Fig. 7 The effect of As(V) adsorption at different pH values

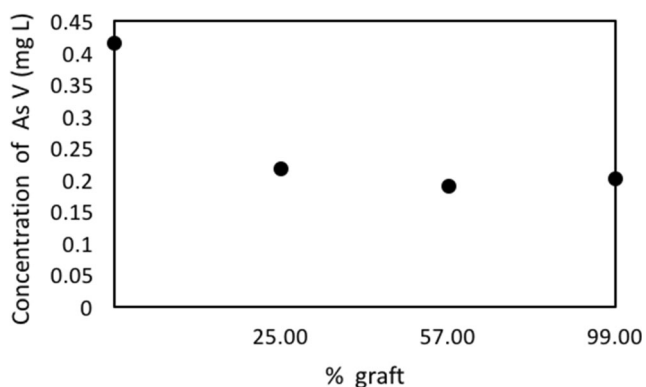


Fig. 8 The effect of As(V) adsorption on the different grafting degrees of PP-Q grafting

h = Constant in the initial range (mg/g h)

a = Initial adsorption rate (g/mg min)

b = Desorption constant (mg/g)

Figure 9 represents the data adjusted to the three models, while Table 2 shows the parameters obtained from the linear regression adjustment of the kinetic data to the models studied. The adsorption process of arsenic ions on the PP-Q membranes was analyzed, evaluating the correlation coefficient and mechanism proposed by the three methods. The first-order model analysis suggests that it is no adjustment considering the values of “ q_e .” However, the pseudo-second-order model presents an excellent adjustment of “ q_e ” and explains that the interaction between PP-Q and arsenic ions corresponded for the chemisorption process. The Elovich model, which is also based on chemical adsorption, did not present a reasonable adjustment since its correlation coefficient is 0.84 and considered low compared with the pseudo-second-order model. These results accord with arsenic adsorption studies reported using chitosan-based adsorbent (Bhowmick et al. 2014; Elwakeel and Guibal 2015; Qi et al. 2015; Malwal and Gopinath 2017; Lee et al. 2017; Asere et al. 2017). In conclusion, the pseudo-second-order model is the

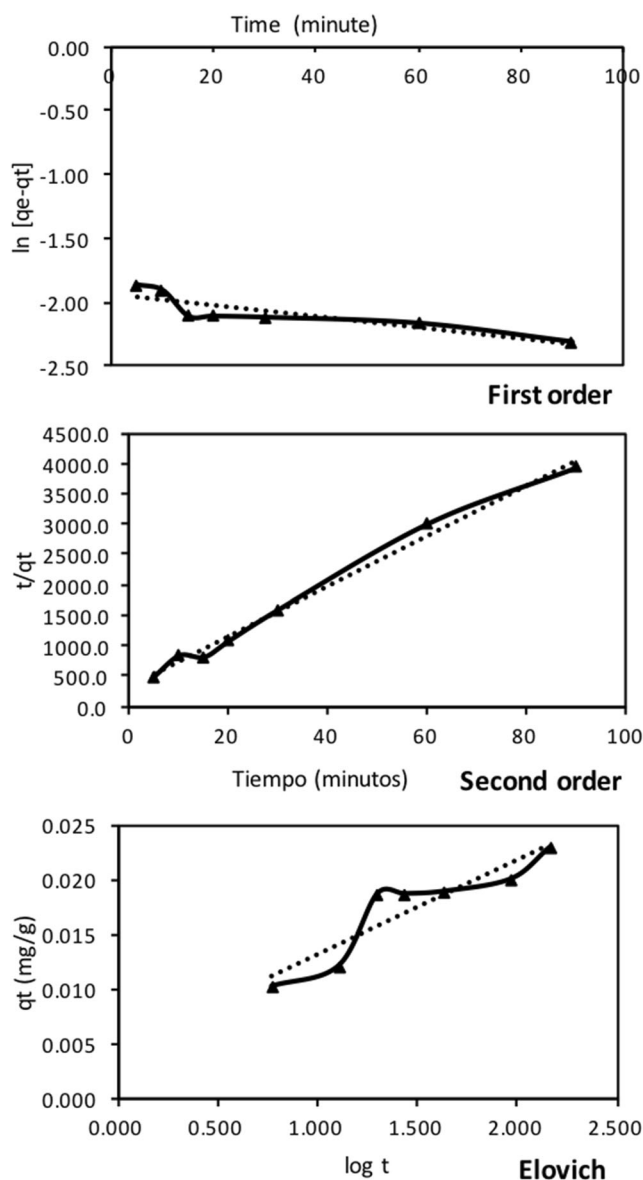


Fig. 9 Kinetics of arsenic sorption on the PP-Q with pseudo-first-order, pseudo-second-order, and Elovich models (25 °C, pH of 1 ± 0.1)

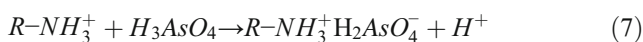
Table 2 Parameters of the kinetic adsorption models of the PP-Q membrane for As(V)

Model	Kinetic constants
Pseudo 1st order	$K_1 = 0.006$ $R^2 = 0.73$ q_e theo. = 0.0113 q_e exp. = 0.031
Pseudo 2nd order	$K_2 = 5.609$ $R^2 = 0.99$ q_e theo. = 0.0240 q_e exp = 0.031
Elovich	$a = 0.0045$ $b = 3.73 \times 10^{-3}$ $R^2 = 0.84$

most appropriate to describe As(V) adsorption using chitosan supported onto a modified polypropylene membrane.

Adsorption mechanism

The analysis of the effect of pH, point of zero charge, the functional groups of chitosan, and speciation of As(V) were used to understand the mechanism of adsorption of As(V) with PP-Q. The pH analysis showed the high removal of As(V) occurs at low pH, whereas the zero-charge point was 7.01. For this reason, the better remotion occurs at a pH below 6.5.



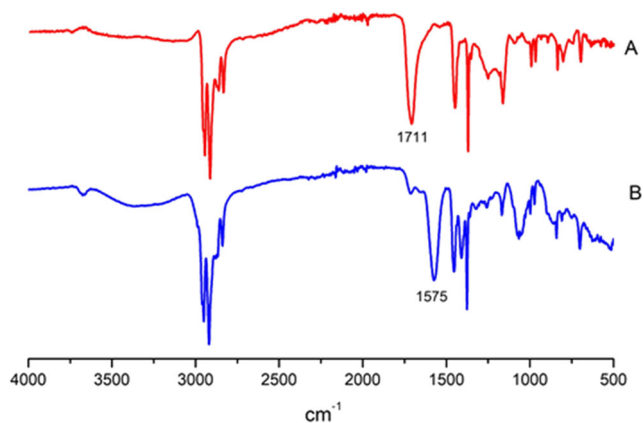


Fig. 10 FT-IR-ATR spectrum of (A) PP-Q and (B) PP-Q-As, after removal of arsenate ions

Therefore, the adsorption occurs by complexation between the functional groups (NH_2) supported on polypropylene (PP-Q) and arsenic ions. Equation 7 represents the adsorption reaction between arsenate ions and free amine groups on the PP-Q membrane's surface. This mechanism was supported by the kinetic study, where the chemisorption process was predicted.

Besides, the IR spectrum also showed changes in the signals due to the interactions between functional groups onto the modified membrane and As(V) solution. In Fig. 10, the signal at 1715 cm^{-1} corresponding to functional groups of chitosan (PP-Q) shifts at 1573 cm^{-1} because of the interaction with arsenic ions (PP-Q-As).

Conclusions

Chitosan supported on modified polypropylene membrane by grafting polymerization reaction was an efficient process. Chitosan did not lose its reactivity to remove the As(V) from the aqueous solution. The adsorption of As(V) increased in contact with the PP-Q at 15 min instead of 90 min. The highest adsorption capacity was obtained at pH 1 (0.031 mg/g), and the removal efficiency of As(V) was 75%. Amine group protonation controlled the chitosan's adsorption mechanism. The membrane PZC was around pH 7. The adsorption of arsenic by polypropylene-chitosan followed the pseudo-second-order kinetic model, suggesting a chemisorption process. Therefore, chitosan-modified membranes have a potential for arsenic (V) remediation.

Nomenclature K_L , pseudo-first-order constant or sorption rate (L/min); q_{es} , amount of solute adsorbed at equilibrium (mg/g); q_t , Amount of solute adsorbed at time t (mg/g); t , time (min); K_2 , pseudo-second-order constant ($\text{g}/\text{mg min}$); h , constant in the initial range ($\text{mg}/\text{g h}$); a , initial adsorption rate ($\text{g}/\text{mg min}$); b , desorption constant (mg/g); q , adsorption capacity (mg/g); v , volume (L); c_i , initial concentration; c_f , equilibrium concentration of As(V) in the solution; m , mass of the dry membrane

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Author contribution José Juan. García-García: experimental development of the research work.

Rosa María Gómez-Espinosa: author of correspondence, development, and coordination of the research.

Reyna Natividad Rangel: in charge of the kinetic study.

Rubí Romero Romero: in charge of the adsorption mechanism.

Gabriela Roa Morales: in charge of speciation studies.

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Data availability Not applicable.

Declarations

Ethics approval Not applicable (there was no need for ethical approval due to the type of study).

Consent to participate. Not applicable.

Consent to publish. Not applicable.

Competing interests The authors declare no competing interests.

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