Semiequilibrium Dialysis versus Ultrafiltration for the Separation of Arsenic from Water Using Cationic Amphiphilic Aggregates

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Abstract

A process, which does not require the addition of large amounts of chemicals, for the removal of arsenic (V) utilizing amphiphilic aggregates (i.e. cetylpyridinium chloride (CPC) micelles) and ultrafiltration (UF) membranes has been studied. The suitability of UF process was evaluated with semi-equilibrium dialysis (SED) cell since the UF process incorporated with amphiphilic aggregates is an equilibrium process. The SED removes up to 100% arsenic (V) from simulated water with the presence of CPC micelles. Furthermore, UF experiments were carried out using regenerated cellulose (RC) membranes (10 kDa NMWCO) and polyethersulfone (PES) membranes (5 and 10 kDa NMWCO) with and without the addition of CPC. Without the addition of CPC, all UF membranes failed to reduce arsenic concentration below the revised maximum contaminant level (MCL) of 10 ppb. However, in the presence of CPC micelles arsenic concentration was reduced below the MCL. The membrane material, membrane nominal molecular weight cut-off (NMWCO) and water pH were found to play an important role on the arsenic removal efficiency.

1. Introduction

It is undeniable that high arsenic concentrations detected in drinking water is a global concern because of its wide toxicological and carcinogen effects to human health [1]. As a result, the maximum contaminant level (MCL) for arsenic in drinking water was reduced from 50 to 10 ppb by the United States Environmental Protection Agency (EPA) and will become mandatory in 2006. This change will affect about 4000 drinking water treatment systems as their arsenic concentrations exceed 10 ppb [2]. It is also estimated that the treatment cost will be higher for rural areas because of their smaller scale [3]. Therefore, new cost effective treatment technologies capable to reduce the arsenic concentration below 10 ppb are needed.

Arsenic in water occurs in two valence states, arsenic (III) species [As(III)] and arsenic (V) species [As(V)]. In natural waters, As(III) species consist primarily of arsenious acid (H₃AsO₃) under reducing conditions, whereas, As(V) species consist primarily of the dominant anionic arsenate ions (H₂AsO₄⁻, HAsO₄²⁻) under oxidizing conditions [4-8]. Arsenic removal is dependent upon the ionic form of arsenic present in water and water chemistry (redox potential (Eh) and pH). The removal efficiency of As(III) is low because it predominantly occurs in the uncharged (H₃AsO₃) state in water.

On the other hand, arsenate anions are more easily removed using conventional separation processes such as chemical precipitation, adsorption and ion exchange. However, these systems produce toxic sludge streams and often are not capable of reducing the final arsenic concentrations to the desirable limit. On the other hand, membrane separation processes can provide extremely low arsenic levels in treated water [9]. Therefore, several studies have been conducted in order to determine the efficiency of microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) for arsenic removal [10-17].

High arsenic removal efficiencies may be obtained using RO and NF membranes (95 and 90%, respectively). However, the water recovery and permeate flux for both membranes are extremely low. In addition, the removal efficiency during the process may drop significantly and they require high operating pressures as they employ relatively dense membranes [11,15]. On the other hand, UF and MF have higher water recovery and permeate flux, and are attractive as a low-energy method. Despite their advantages, UF is still not a feasible option as it only removes about 47% of arsenate ions [10]. Arsenic rejection increases to 70 % if the membrane surface is negatively charged and the water has high dissolved organic carbon [15,16]. MF removes only the particulate form of arsenic due to its large pore size [11].

Limitations in the conventional removal processes urge for new alternative technologies. UF can be an alternative technology if used in combination with amphiphilic molecules to bind the contaminants from a water stream. This technique has high flux rates, generally is a low-energy, low-pressure process and is cost effective, since it employs loose membranes. When an ionic amphiphile is placed in aqueous solution in concentrations beyond its critical micellar concentration (CMC), amphiphilic aggregates (micelles) are formed. The surface of the amphiphilic aggregates has a high charge density and a high absolute electrostatic potential; thus multivalent cations/anions in the aqueous phase electrostatically can bind onto the micellar surface. The resultant colloid is physically too large to pass through membrane pores, and is retained on the concentrate side (Fig. 1). Previous studies have shown the potential of this technique for removing heavy metals from water streams [18-27]. This process is found to be an equilibrium controlled process rather than a kinetically controlled process [28].



Fig. 1. Schematic of experimental water treatment process.

The objective of this study is to develop an effective treatment method for the removal of As(V) from water using UF membranes [regenerated cellulose (RC) and polyethersulfone (PES) membranes] and cationic cetylpyridinium chloride (CPC). The efficiency of the UF process and the concentrations of

CPC and As(V) that would be expected to transfer through UF membranes under near-equilibrium conditions were pre-determined using the Semi-Equilibrium Dialysis (SED) technique [28] since this process is an equilibrium controlled process. The UF process parameters for optimum separation was then determined by investigating process parameters such as feed water As(V) concentration, water pH, membrane NMWCO, and feed water amphiphilic molecule concentration.

2. Experimental

2.1. Materials

Analytical reagent grade arsenic pentoxide (As₂O₅, 99% purity) was obtained from Alfa Aesar (Ward Hill, MA). It forms H_3AsO_4 in water [30]. Cetylpyridinium chloride (CPC) $[C_{16}H_{33}(N(C_5H_5))^+Cl^-]$ with a purity of 100% was provided by Zeeland Chemicals (Zeeland, MI). These chemicals were used as received. Certified standard sodium hydroxide (1 N), obtained from Fisher Scientific, was used for pH adjustments. The water was distilled and double deionized (DDI).

The flat sheet UF membranes used in this study were regenerated cellulose (RC) membranes with a 10 kilo-Dalton (kDa) NMWCO and polyethersulfone (PES) membranes with a 5 kDa and 10 kDa NMWCO (Millipore, Bedford, MA). Prior to the experiments, the membranes were soaked in DDI water for 24 hours.

2.2. Methods

Glasswares were pre-cleaned using a regular lab detergent, acid washed with Nochromix solution (Godax Laboratories Inc., Takoma Park, MD) and well rinsed with DDI water. Concentrated stock solutions for CPC (0.1 M) and As_2O_5 [22,980 ppb as As (V)] were prepared with DDI water and diluted to the lower concentrations. The stock solution of As (V) was refrigerated (4 °C) for a month in order to ensure the formation of H₃AsO₄. The type and charge of the arsenic species [8] in feed water were determined by measuring the pH and oxidative reduction potential (Eh) levels with an OAKTON model 2500 series Digital pH meter (OAKTON Instruments, Vernon Hills, IL). For all experiments, the measured Eh values of the feed, retentate, and permeate solutions at pH 5.5 and 8 assured the existence of the mono-anionic form of As (V) and the di-anionic form of As (V), respectively.

2.2.1. SED Experiments

Ordinary 5 mL equilibrium dialysis cells and regenerated cellulose dialysis membranes (6 kDa molecular weight cut-off) were obtained from Fisher Scientific. Prior to the experiments, the membranes were soaked in DDI water for 24 hours. Aqueous solutions of CPC (10, 50 and 100 mM) or of CPC (100 mM) plus As(V) (15 - 442,975 ppb) were placed on one side of the SED compartment (retentate side) and DDI water was added to the other side (permeate side). The SED cells were then equilibrated at room temperature for 24 hours. After which, samples from both the retentate and permeate sides were withdrawn and analyzed for As (V) and CPC as explained in section 2.2.3.

2.2.2. UF Experiments

Simulated feed water, prepared from secondary stock solutions of CPC and As(V), was used in the ultrafiltration experiments. The concentration of CPC in feed water was either 0 mM or 10 mM. The concentration of As (V) in feed water was varied between 0 ppb and 221 ppb.

Ultrafiltration experiments were carried out at room temperature in a 400 mL batch-stirred cell unit (Spectrum Laboratories Inc., Rancho Dominguez, CA). The UF cell was initially filled with 300 mL feed water and the pressure gradient across the membrane was maintained at 414 kPa with UHP nitrogen gas. The magnetic stirrer speed was kept at 850 rpm. Permeate water samples were collected for As (V) and CPC analyses. Each experimental run was terminated when 90% of the feed water was filtered. The effective surface area of the membrane, which was exposed to the feed solution during the UF experiment, was $38.5 \times 10^{-4} \text{ m}^2$ [29].

2.2.3. Sample Analysis

The concentrations of As(V) in feed, permeate, and retentate water samples collected from the experiments performed without amphiphilic molecules were determined according to the standard method of EPA 200.8 [31]. The Sciex Elan 5000 Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (Perkin Elmer, Norwalk, CT) was used in conjunction with Perkin Elmer AS-91 autosampler and peristaltic pump. The mean of three concentration values with its standard deviation was reported for each sample.

The concentrations of As(V) in feed and retentate water samples collected from the experiments performed with amphiphilic molecules were determined by using the standard method of EPA 200.7 [32]. The Perkin Elmer Optima 3000 DV Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) (Perkin Elmer, Norwalk, CT) was used. The permeate water samples of these experiments were analyzed using the standard method of EPA 200.8 as described above. All water samples were analyzed without dilution. The mean of three concentration values with its standard deviation was reported for each sample.

The concentration of CPC in feed, permeate, and retentate water samples was determined using a diode array UV-vis 8453 spectrophotometer (Agilent, Wilmington, DE) at a wavelength of 259 nm.

A feed-based As (V) and CPC rejections as percentage are defined as:

Feed – Based As (V) or CPC rejection,
$$(\%) = \left(1 - \frac{C_{A,permeate}}{C_{A,feed}}\right) x100$$
 (1)

where $C_{A,permeate}$ and $C_{A,feed}$ are the concentrations of As (V) or CPC in the permeate and feed water samples, respectively.

The binding capacity of CPC micelles $q_e (mg/g)$ at equilibrium was calculated as:

$$q_e = \frac{C_{As(V)}, _{retentate} - C_{As(V), permeate}}{(C_{CPC}, _{retentate} - C_{CPC, permeate}) x M W_{CPC}}$$
(2)

where As(V) concentrations are in mg/L, CPC concentrations in M, and MW_{CPC} is the molecular weight of CPC. To describe the binding behavior, the well known Langmuir and Freundlich expressions were used,

Langmuir:
$$qe = \frac{q_o K_L C_{As(V), permeate}}{1 + K_L C_{As(V), permeate}}$$
 (3)

Freundlich:
$$qe = K_f C_{As(V), permeate}^{1/n}$$
 (4)

where $q_o \text{ (mg/g)}$ is the maximum amount of As(V) bound per unit weight of CPC micelle, K_L is the affinity of binding sites (L/mg), K_f and 1/n are roughly indicators of the binding capacity and of the binding intensity, respectively. These parameters are computed from the slopes and intercepts of the linearized form of the Langmuir and Freundlich expressions.

3. Results and discussion

3.1. SED Analysis

The results of SED experiments for aqueous solutions of CPC only, As(V) only, and CPC containing known concentrations of As(V) for a water pH of 5.5 and 8 are presented in Table 1. As can be seen in Table 1, the total concentration of CPC in permeate is around its CMC value of 0.00088 M indicating that CPC micelles do not pass through the dialysis membrane in significant amounts. The results of SED experiments show that some As(V) rejection is obtained in the absence of CPC micelles due to the mechanism of Donnan exclusion [33]; electrostatic rejection of ions due to a net charge of membrane above its isoelectric point. RC membrane has an isoelectric point at about 3.5 and the negative charge dominates on the membrane surface at a higher pH [34]. Thus, arsenic rejection increases as the pH increases from 5.5 to 8 due to the net increase in membrane charge and the transition of As (V) from mono-anionic to di-anionic form (Table 1).

	initial		permeate		retentate		% rejection	
pН	[CPC], mM	As(V), ppb	[CPC], mM	As(V), ppb	[CPC], mM	As(V), ppb	CPC	As(V)
5.5	9.9	-	0.82	-	7.9	-	91.7	-
	48.9	-	0.87	-	42.3	-	98.2	-
	98.0	-	0.97	-	74.9	-	99.0	-
8	10.1	-	0.81	-	8.4	-	92.0	-
	50.5	-	0.70	-	43.9	-	98.6	-
	101.1	-	0.64	-	79.6	-	99.4	-
5.5	-	15	_	4	-	8	-	73 3
	-	41	-	12	-	25	-	71.4
	-	100	-	23	-	70	-	76.7
	-	999	-	328	-	622	-	67.2
	-	8384	-	2979	-	4967	-	64.5
8	-	15	-	4	-	9	-	75.9
	-	44	-	10	-	27	-	76.8
	-	100	-	26	-	72	-	73.5
	-	999	-	280	-	653	-	72.0
	-	8623	-	2657	-	5094	-	69.2
8	109.9	15	1.00	0	99.0	15	99 1	100.0
	98.6	25	1.00	0	99.0	25	99.0	100.0
	102.6	50	1.00	0	99.0	50	99.0	100.0
	109.2	100	1.00	0	99.0	99	99.1	100.0
	110.1	9990	0.99	122	99.0	9870	99.1	98.8
	97.8	99895	0.90	3516	99.1	96380	99.1	96.5
	104.2	172304	0.87	13068	84.9	121580	99.2	92.4
	107.1	214706	0.76	18930	87.4	158550	99.3	91.2
	106.4	292770	0.67	26991	87.6	204900	99.4	90.8
	105.6	442975	0.58	51022	86.4	296960	99.4	88.5

Table 1. Semi-equilibrium dialysis data for the ternary system CPC/As(V)/water.

The highest arsenic rejection obtained without CPC micelles turned out to be 76.8%. With CPC micelles, the arsenic rejection was found to be 100% for lower initial arsenic concentrations and 88.5% for the highest initial arsenic concentration studied. The favorable exchange of As(V) ions with the counter ion chloride is based on electrostatic binding of the highly negatively charged As(V) anions to the positively charged pyridinium head groups of the micelle. The decrease in arsenic rejection at very high initial As(V) concentrations could be due to the consumption of available binding sites and disintegration or structural changes of micelles at such high As(V) concentrations. In any case, the use of micelles show that very large separation factors can be obtained for low initial As(V) concentrations. Thus, for trace amount of contamination, where enough micellar binding sites are available for As(V) ions, practically all of the As(V) ions can be removed from water when suitable micelles and UF membranes are used.

The equilibrium data for CPC-As(V) systems, shown in Table 1, were also analyzed using the Langmuir and Freundlich isotherms. The linearized plot of the Langmuir ($1/q_e$ versus $1/C_{As(V),permeate}$) and Freundlich (log q_e versus log $C_{As(V),permeate}$) models are shown in Fig. 2 and 3. Calculated parameters of these models are reported in Table 2. As can be seen from Figs. 2 and 3, the equilibrium data fits very well to both the Langmuir and Freundlich expression with very high correlation coefficients of 1.0 and 0.9885, respectively, confirming the applicability of both the models. The calculated Langmuir and Freundlich constants q_o , K_L , K_f and n, are 8.64 mg/g, 0.26 L/mg, 0.54 (mg/L)ⁿ and 1.3, respectively. The higher adsorption capacity $(q_o > 1)$ and binding intensity (n > 1) confirms that CPC micelles can be effectively used for the removal of As(V) from its aqueous solutions.



Fig. 3. Equilibrium data for linearized Freundlich isotherm.

-1

log CAs(V), permeate

0

1

2

3

-2

-3

-5

-4

	¥		Langmuir	Fre	undlich	1	
рН	Amphiphile	q _o (mg/g)	K _L (L/mg)	R^2	$K_f(mg/L)^n$	n	R^2
8	CPC	8.64	0.26	1	0.54	1.3	0.9885

Table 2. Langmuir and Freundlich constants for CPC-As(V) systems.

3.2. UF Experiments

In order to verify the As(V) removal obtained through SED analysis, the UF experiments were conducted using different types of UF membranes without and with the addition of CPC micelles. Fig. 4 shows the As(V) removal without the addition of CPC micelles using different types of UF membranes. Lower arsenic removal efficiencies are obtained with PES membranes (up to 12.2%) due to its higher isoelectric point. Higher arsenic removals were obtained with RC membranes (up to 70.7%) owing to their negative surface charge [29,34]. The increase in water pH further increases the arsenic removal with RC membranes due to the net increase in the membrane surface charge and the transition of As (V) from mono-anionic to di-anionic form (Fig. 4). However, none of the UF experiments performed in the absence of CPC could produce permeate arsenic concentrations lower than the permissible limit of 10 ppb. The data obtained with SED analysis is consistent with the data obtained using a batch UF unit. The difference in As(V) removal can be attributed to the difference in the NMWCO used for the RC membrane.

As seen in Fig. 5, arsenic removal significantly increases with the addition of CPC (10mM) and was found to be between 90.9% and 100%. The significant increase in arsenic removal confirms the SED analysis. As expected, cationic CPC micelles effectively bind the oppositely charged As(V) anions and the resultant colloids are then retained by the PES and RC membranes.

Arsenic removal was highest (100%) with 5 kDa PES membrane (at pH 8) and lowest (90.9%) with 10 kDa PES membrane (at pH 5.5) (Fig. 5). In general, arsenic removal efficiency decreases slightly as the feed water arsenic concentration increases (Table 1, Figs. 4 and 5).



Fig. 4. As(V) removal with UF membranes as a function of feed water arsenic concentration and feed water pH in the absence of CPC micelles (reproduced with permission from reference 29).



Fig. 5. As(V) removal with UF membranes as a function of feed water arsenic concentration and feed water pH in the presence of CPC micelles (reproduced with permission from reference 29).

The permeate arsenic concentration will determine the suitability of the treated water for drinking. In particular, the concentrations of unbound As (V) in the permeate water obtained from the SED experiments are practically the same as that obtained from the UF experiments. Slight differences can be attributed to the different types of membranes used for UF and SED analysis. As seen in Fig. 6,

for all but one UF experiment, permeate arsenic concentrations fall below the new MCL of 10 ppb and are in good agreement with SED analysis. Regardless of the initial arsenic feed concentration, permeate arsenic concentrations fell well below the ICP-MS detection limit using 5 kDa PES membranes at a solution pH of 8 making it our optimum selection.

As seen in Fig. 6, the arsenic concentrations in permeate water increase linearly with increased feed water arsenic concentration. This is due to the ion shielding of the effective charge of the membrane and the consumption of available binding sites on the micellar surface [29]. Furthermore, the slope of linearity decreases with increased pH (less arsenic in the permeate) (Fig. 6). In addition, the same trend was observed for arsenic concentrations in retentate water. Unlike permeate data, the slope of linearity increases with increased pH (more arsenic in the retentate) (Fig. 6).



Fig. 6. As(V) concentrations in permeate and retentate water as a function of feed water arsenic concentration, membrane type, and feed water pH (reproduced with permission from reference 29).

Although the removal of arsenic using CPC micelles and UF membranes is highly efficient, there is one major drawback. The micelle formation is dynamic and the monomeric units that exist in equilibrium are roughly equal to the CMC. These monomers may pass through the UF membrane as the feed water is filtered. Hence, the concentration of CPC in permeate is expected to be around the CMC of CPC (0.88 mM) [29]. However, our studies have shown that the concentration of CPC in permeate is also affected by the membrane characteristics and water pH levels. Smaller NMWCO membranes resulted in lower CPC concentration in permeate. The permeate CPC concentrations for 5 kDa PES, 10 kDa PES and 10 kDa RC were measured around 0.2 mM (pH = 5.5) and 0.13 mM (pH = 8), 1.20 mM (pH = 5.5) and 1.08 mM (pH = 8), 0.93 mM (pH = 5.5) and 0.86 mM (pH = 8), respectively, and did not change with the initial arsenic concentration.

Conclusion

Without CPC micelles, all UF membranes failed to reduce the permeate arsenic concentrations below the revised MCL of 10 ppb. Owing to their negative surface charge, RC membranes provided better arsenic removal than PES membranes. On the other hand, in the presence of CPC micelles, all UF membranes reduced the permeate arsenic concentrations below the MCL. It was concluded that selection of the membrane material, membrane NMWCO, and water pH play an important role on the arsenic removal efficiency. Regardless of the feed water arsenic concentration, 100% arsenic removal was obtained with 5 kDa PES membranes at pH of 8 making it our best choice for UF. The same removal efficiency was also achieved for the feed water arsenic concentrations of 22 and 43 ppb using 5 kDa PES membranes at pH of 8.

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