

Removal of Arsenic from Water Using Amphiphilic Molecules and Ultrafiltration Membranes

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Abstract

Arsenic (V) removal using cetylpyridinium chloride (CPC) micelles and 5 kilo-Dalton (kDa) polyethersulfone (PES) membrane was studied in the presence of multiple co-ions. The concentrations of arsenic ($[\text{As}]_F = 0 - 105 \mu\text{g/L}$) and co-ions ($[\text{CO}_3^{2-}] = 0 - 4 \text{ mg/L}$, $[\text{PO}_4^{3-}] = 0 - 0.3 \text{ mg/L}$, $[\text{SiO}_3^{2-}] = 0 - 71 \text{ mg/L}$, and $[\text{SO}_4^{2-}] = 0 - 400 \text{ mg/L}$) in simulated feed water were varied. Without the addition of CPC micelles, the PES membrane was found to be ineffective for arsenic removal. In the presence of co-ions, the highest arsenic removal was 25%. However, the addition of CPC micelles significantly increases the arsenic removal efficiency. Regardless of the presence of co-ions, the arsenic concentrations in permeate water were consistently reduced well below the new maximum contaminant level (MCL) of $10 \mu\text{g/L}$. The repeatable results obtained suggest that using ultrafiltration membranes with CPC micelles can be an alternative treatment technology for arsenic removal from contaminated water.

1. Introduction

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) due to its wide toxicological and carcinogen effects [1,2]. Consequently, all public water suppliers are required to maintain arsenic concentrations at or below the new MCL by 2006. It is estimated that about 4000 community water systems may require additional treatment technologies in order to comply with the revised MCL [2]. In addition, the treatment costs are expected to be higher for rural areas due to their smaller scale operations [3]. Therefore, new cost-effective alternative treatment technologies are required.

Arsenic exists in a variety of inorganic forms and oxidation states in water. The pentavalent form, arsenic (V) species $[\text{As(V)}]$, is generally found in surface water due to oxidizing conditions. The trivalent form, arsenic (III) species $[\text{As(III)}]$, is generally found in ground water due to reducing conditions. As(III) species may be present as arsenious acid (H_3AsO_3) and arsenite ions (H_2AsO_3^- ,

HAsO_3^{2-} , and AsO_3^{3-}). Similarly, As(V) species may exist as arsenic acid (H_3AsO_4) and arsenate ions (H_2AsO_4^- , HAsO_4^{2-} , AsO_4^{3-}) [4-9]. The transitions between As(III) and As(V) oxyanions can be controlled by the water pH and the oxidative reduction potential (Eh). The Eh-pH relationship for the As-O-H system at 25 °C and 1 bar can be found in references 8 (as figure) and 9 (as empirical correlations). The ionic form of arsenic affects the efficiency of treatment methods. Because of its ionic charge, As(V) is easier to remove from source waters than As(III) using conventional separation processes (precipitation, ion exchange, sorption). However, these systems may require secondary treatment systems to reduce the arsenic concentration below the new MCL. Because membrane separation processes have the potential to provide extremely low arsenic level in treated water [10], several researchers have tested microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) as an alternative arsenic removal technology [11-18].

NF is capable of removing arsenic up to 90% but with only 15% water recovery. RO provides about 95% arsenic removal efficiency with 10 to 50% water recovery. However, the final water stream is deficient in minerals. In NF systems, water can be recovered up to 90%, but the removal efficiency reduces to 16%. Both systems, RO and NF, use relatively dense membranes which have very low permeability. Thus, higher operating pressures and feed quality waters are required [12, 16]. In the case of UF and MF, the water recovery is much higher than that of NF and RO because they use less dense or loose membranes. Additionally, both UF and MF provide high fluxes at low temperature and low pressure, making them low-energy methods. However, UF was found to remove only 47% of As(V) [11]. Arsenic removal can be increased to 70% if the membrane surface is negatively charged [9] and the water has high dissolved organic carbon [16, 17]. On the other hand, MF removes only the particulate form of arsenic due to the larger pore sizes [12].

New alternative technologies for the removal of dissolved arsenic species are in demand due to limitations in the conventional removal processes. In our previous work, UF membranes were found to be efficient for arsenic removal when used in combination with cationic micelles [9]. This technique has also proved effective for the removal of heavy metals from water [19-29]. In this method, the cationic micelles that electrostatically bind anionic arsenic species are mixed with a contaminated drinking water stream. The aggregates with bound arsenic ions are large enough to be retained by a UF membrane. It was found that this technique could achieve 100% arsenic removal with high flux rates. However, the removal of arsenic in the presence of co-ions has not yet been investigated using this technique.

This work investigates the effect of co-ions (CO_3^{2-} , PO_4^{3-} , SiO_3^{2-} , and SO_4^{2-}) on arsenic removal efficiency by using CPC micelles and PES membranes. The feed water condition in terms of co-ion concentration and As(V) concentration were varied, and the efficiency of the process was tested using simulated water samples.

2. Experimental

2.1. Materials

Arsenic (V) oxide (As_2O_5) (99% purity) was obtained from Alfa Aesar (Ward Hill, MA). It forms H_3AsO_4 in water [30]. The cationic amphiphile cetylpyridinium chloride (CPC) [$\text{C}_{16}\text{H}^+_{33}(\text{N}(\text{C}_5\text{H}_5))^+\text{Cl}^-$] (100% purity) was provided by Zeeland Chemicals (Zeeland, MI). Sodium carbonate (Na_2CO_3), sodium phosphate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$), sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$), and sodium sulfate (Na_2SO_4) were certified A.C.S. grade and purchased from Fisher Scientific. Certified standard

sodium hydroxide (1 N NaOH), obtained from Fisher Scientific, was used for pH adjustment. All chemicals were used without purification. Stock solutions and simulated water were prepared using double distilled deionized (DDI or ultra clean) water.

Flat sheet hydrophilic PES membranes of 5 kilo-Dalton nominal molecular weight cut off (NMWCO) were purchased from Millipore, Bedford, MA. These membranes were conditioned in DDI water for 24 hours prior to the experiments.

2.2. Methods

The simulated feed water was prepared by using less concentrated secondary stock solutions of As (V) (22,980 ppb), CPC (0.1 M), Na₂CO₃ (566 ppm as CO₃⁻²) and Na₃PO₄·12H₂O (500 ppm as PO₄⁻³). On the other hand, Na₂SO₄ or Na₂SiO₃·9H₂O salt was directly weighted and added to the simulated feed water to achieve the desired concentration.

The pH of the simulated feed water was adjusted to 8 using certified standard 1 N NaOH solution. The pH was verified using an OAKTON model 2500 series Digital pH/ion/ORP/°C Meter (OAKTON Instruments, Vernon Hills, IL).

2.2.1. UF Experiments

UF experiments were performed with simulated feed water. The arsenic concentration in simulated water was varied between 0 and 105 ppb. In addition, it contains co-ions ([CO₃⁻²] = 0.15, 1.35, or 4 ppm, [PO₄⁻³] = 0.05, 0.15, or 0.3 ppm, [SiO₃⁻²] = 5, 25, or 71 ppm, and [SO₄⁻²] = 28, 100, or 400 ppm) and CPC (0 or 10 mM). The co-ion concentrations were obtained from the water quality database supplied by the seventeen US water utility companies [31]. They correspond to the low, average, and high concentration levels of these co-ions in water.

UF experiments were performed at room temperature as described in our earlier study [9]. Following the UF experiments the exact concentrations of As (V) and CPC in feed, retentate, and permeate water samples were determined with the analytical methods discussed in section 2.2.2. The pH and oxidative reduction potential (Eh) of water samples were measured with an OAKTON model 2500 series Digital pH/ion/ORP/°C meter (OAKTON Instruments, Vernon Hills, IL) to verify the type and charge of arsenic species in water samples.

The effective surface area of the membrane, which was exposed to the feed solution during the UF experiment, was 38.5x10⁻⁴ m².

For evaluation purposes a feed-based arsenic removal percentage was calculated from:

$$\text{Feed - Based Arsenic removal (\%)} = \left(1 - \frac{C_{A,permeate}}{C_{A,feed}} \right) \times 100 \quad (1)$$

where $C_{A,permeate}$ and $C_{A,feed}$ are the concentrations of arsenic in the permeate and feed water samples, respectively.

2.2.2. Sample Analysis

The exact concentration of As(V) in feed, permeate, and retentate water samples was determined according to the standard method of EPA 200.8 [32] using a Sciex Elan 5000 Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (Perkin Elmer, Norwalk, CT) in conjunction with Perkin Elmer AS-91 auto sampler and peristaltic pump. In this study, all quality control checks were within 10% of the true value and the mean of the three intensity values was reported.

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

3. Results and Discussion

3.1. Arsenic Removal with UF Membrane

Table 1 shows the experimental results in terms of As(V) concentration in the feed and permeate water samples, Eh values, percent arsenic removal, and the co-ion concentration in the feed water. The Eh values for all water samples (between -150 mV and 758 mV [9]) assure the existence of As(V) in di-anionic form at pH 8.

Table 1. UF experiments performed with simulated water and PES membranes (pH=8, [CPC]=0 mM). As is arsenic (V). F, R, and P represent feed, retentate, and permeate, respectively.

Ion	[Ion] (ppm)	Concentration (ppb)		Eh (mV)			Removal (%)
		[As] _F	[As] _P	F	R	P	F-Based
None	0	41	36	223.8	221.4	227.9	12.2
		79	73	216.5	236.1	218.5	7.6
CO ₃ ⁻²	1.35	40	35	238.5	242.6	228.6	12.5
		82	65	224.1	239.5	219.9	20.7
PO ₄ ⁻³	0.15	40	31	256.9	226.9	225.8	22.5
		82	65	273.8	233.1	223.5	20.7
SiO ₃ ⁻²	25	40	30	246.8	220.6	214.1	25.0
		82	71	261.3	234	223.8	13.4
SO ₄ ⁻²	100	40	35	266.4	227.5	208.1	12.5
		82	76	251.7	221.8	213.9	7.3

For feed water containing 41 ppb and 79 ppb As(V), the arsenic removal in the absence of co-ions was found to be 12.2% and 7.6%, respectively. Regardless of the type and concentration of co-ions ([CO₃⁻²] = 1.35 ppm, [PO₄⁻³] = 0.15 ppm, [SiO₃⁻²] = 25 ppm, and [SO₄⁻²] = 100 ppm), the highest arsenic removal is 25%. The mechanism of Donnan exclusion, the electrostatic rejection of ions due to a net charge of membrane [33], accounts for the observed arsenic removal. In our previous work, it has been shown that the form of the arsenic anions, membrane surface charge, and the interaction of arsenic with

membrane charge sites affect the overall arsenic removal [9]. The surface charge can be determined with a streaming potential measurement [34] and PES membranes were reported to have a slight negative surface charge at the pH studied [35]. Thus, certain level of arsenic removal is provided by the presence of weak electrostatic repulsion (Table 1).

Increased feed water arsenic concentration and the presence of co-ions (PO_4^{-3} , SiO_3^{-2} , and SO_4^{-2}), decreases arsenic removal. The decrease is most affected by SiO_3^{-2} ions and least affected by PO_4^{-3} ions. Conversely, the presence of CO_3^{-2} ions and an increase in feed water arsenic concentration increases the arsenic removal. All the UF experiments performed in the absence of CPC resulted in the arsenic concentrations in permeate water being higher than the permissible limit of 10 ppb (Table 1).

3.2. Arsenic Removal with CPC and UF Membrane

Table 2 lists the experimental measurements and Figs. 1 through 4 show the effects of CPC, co-ion concentration, and feed water arsenic concentration on the arsenic removal. From Table 2, the existence of As (V) in di-anionic form at pH 8 is verified with Eh values being between 195.7 mV and 311.4 mV for all water samples.

The use of CPC significantly increases the arsenic removal, as can be seen in Figs. 1 through 4. Compared to the highest removal without CPC (25%, Table 1), arsenic removal with CPC was found to be between 78.1% and 100%. These results clearly indicate that the cationic micelles can effectively bind As(V) anions. The resultant colloid is large enough to be retained by the PES membranes, yielding high arsenic removals.

With one exception, the arsenic removal in the presence of CO_3^{-2} , PO_4^{-3} , and SiO_3^{-2} , regardless of the initial arsenic and co-ion concentrations, turned out to be 100%. The aforementioned exception ($[\text{As (V)}] = 105 \text{ ppb}$ and $[\text{SiO}_3^{-2}] = 71 \text{ ppm}$) resulted in 99% removal. This is shown in Figs. 1 through 3 and in Table 2. Arsenic removal in the presence of SO_4^{-2} ions was found to be 100% for the low initial arsenic and SO_4^{-2} concentrations, but was found to decrease to 78% with increased feed water arsenic and SO_4^{-2} concentrations. This is shown in Fig. 4 and Table 2. Three phenomena can be attributed to the significant decrease in the arsenic removal at very high SO_4^{-2} concentrations; the disintegration of CPC micelles, the consumption of the available binding sites of CPC micelles, and ion shielding of the effective charge of the membrane. Cumulatively, the effect is a reduction in arsenic removals.

Arsenic concentration in permeate water plays an important role in determining the suitability of the treated water to be used for drinking water. From Table 2, it can be seen that the PES membrane with CPC, even in the presence of CO_3^{-2} , PO_4^{-3} , SiO_3^{-2} , and SO_4^{-2} , achieves a very high degree of arsenic removal. With a single exception ($[\text{SO}_4^{-2}] = 400 \text{ ppm}$ and $[\text{As}] = 105 \text{ ppb}$), all the UF experiments resulted in permeate water arsenic concentrations below the new MCL of 10 ppb.

Table 2. UF experiments performed with simulated water and PES membranes (pH=8, [CPC]=10 mM). As is arsenic (V). F, R, and P represent feed, retentate, and permeate, respectively.

Ion	[Ion] (ppm)	Concentration (mM)			Concentration (ppb)		Eh (mV)		
		[CPC] _F	[CPC] _R	[CPC] _P	[As] _F	[As] _P	F	R	P
None	0	10.35	81.66	0.15	22	0	268.9	261.9	216.9
		10.11	83.98	0.12	43	0	277.2	272.3	221.3
		10.29	80.09	0.14	83	0	273.4	261.3	211.5
CO ₃ ⁻²	0.15	10.18	86.24	0.16	30	0	280.4	289.7	236.8
		10.54	81.02	0.16	55	0	252.3	251.5	227.5
		10.31	88.11	0.16	105	0	267.5	270.4	218.1
	1.35	10.41	90.35	0.17	30	0	272.1	254.4	240.5
		10.39	90.71	0.17	55	0	301.5	263.5	226.7
		10.21	83.86	0.18	105	0	245.7	231.7	218.4
	4	10.58	93.17	0.17	30	0	238.5	253.1	223.5
		10.39	87.03	0.17	55	0	298.3	272	230.3
		10.42	84.67	0.19	105	0	270.9	239.8	229.1
PO ₄ ⁻³	0.05	10.20	92.66	0.18	30	0	284.8	274.2	243.6
		10.44	88.28	0.17	55	0	267.4	256.7	252.2
		10.46	91.74	0.18	105	0	298.2	246.1	223.2
	0.15	10.37	90.22	0.18	30	0	311.4	297.4	256.8
		10.34	90.78	0.18	55	0	269.8	268.9	243.9
		10.29	87.90	0.17	105	0	283	234.2	226.1
	0.3	10.29	92.29	0.17	30	0	295.7	272.1	237.9
		10.19	91.88	0.16	55	0	248.3	223.5	211.7
		10.27	92.71	0.16	105	0	264	239.2	225.6
SiO ₃ ⁻²	5	10.08	86.21	0.18	30	0	229.4	228.1	207.1
		10.22	91.62	0.18	55	0	238.1	223.4	219.8
		10.10	83.06	0.17	105	0	224.5	225.6	195.7
	25	10.21	88.31	0.17	30	0	246.1	237.1	211.2
		10.40	91.65	0.17	55	0	280.2	267.3	236.8
		10.19	89.72	0.16	105	0	238.5	239.2	221.5
	71	10.52	91.72	0.19	30	0	237.2	225.9	208.9
		10.31	90.94	0.17	55	0	266.1	246.7	222.7
		10.28	92.66	0.17	105	1	224.9	213.9	187.2
SO ₄ ⁻²	28	10.24	87.40	0.16	30	0	251.3	244.3	215.8
		10.87	88.87	0.16	55	0	267.4	271.6	234.9
		10.44	88.48	0.13	105	2	249	243.5	227.6
	100	10.45	86.23	0.16	30	0	242.1	231.4	214.7
		10.52	88.18	0.16	55	1	272.9	253.3	238.3
		10.31	90.03	0.16	105	10	276.9	276.2	242.1
	400	10.58	90.35	0.17	30	0	258.2	249.4	232.5
		10.45	88.97	0.16	55	1	232.7	241	226.1
		10.50	92.92	0.15	105	23	263.8	248.8	253.4

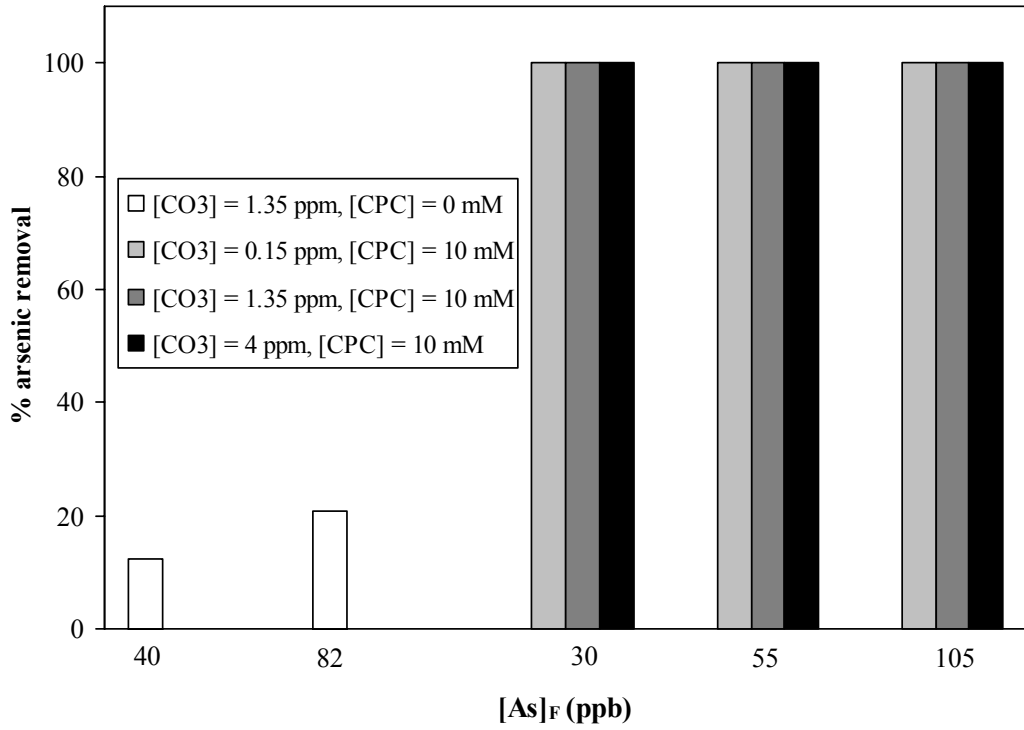


Fig. 1. Effect of CO_3^{2-} , feed water arsenic and CPC concentration on As (V) removal.

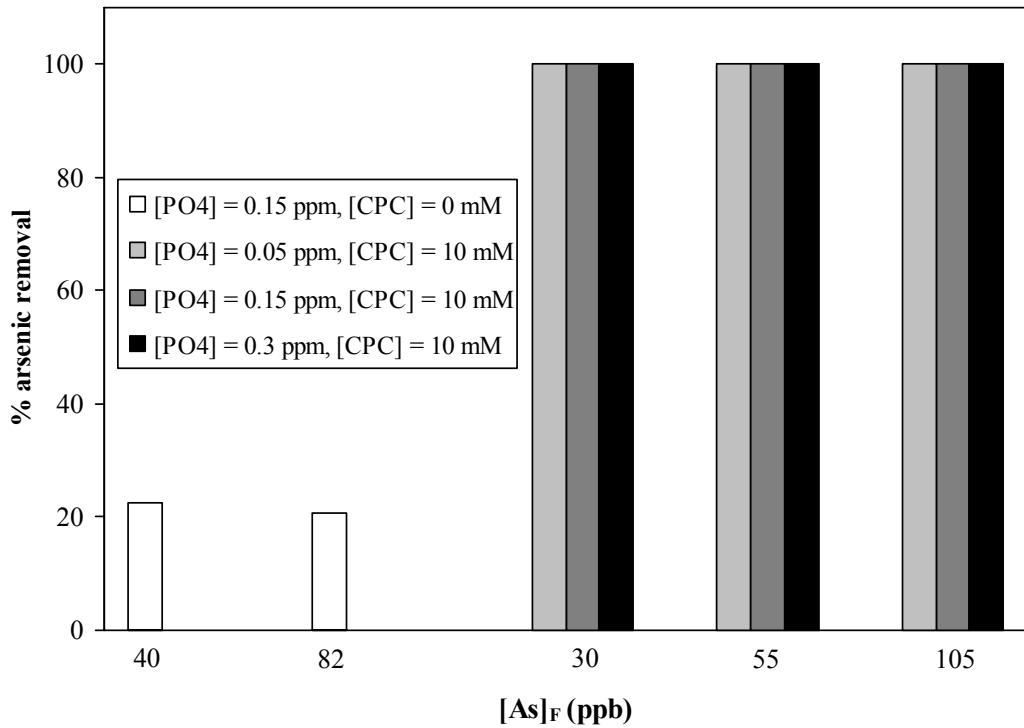


Fig. 2. Effect of PO_4^{3-} , feed water arsenic and CPC concentration on As (V) removal.

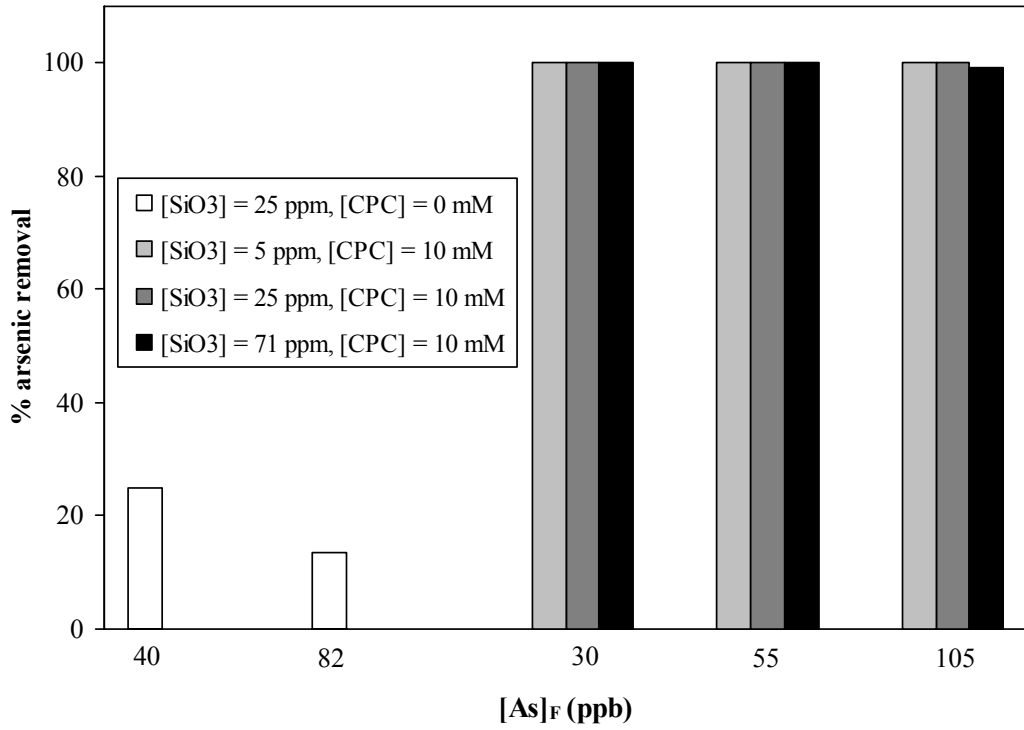


Fig. 3. Effect of SiO₃²⁻, feed water arsenic and CPC concentration on As (V) removal.

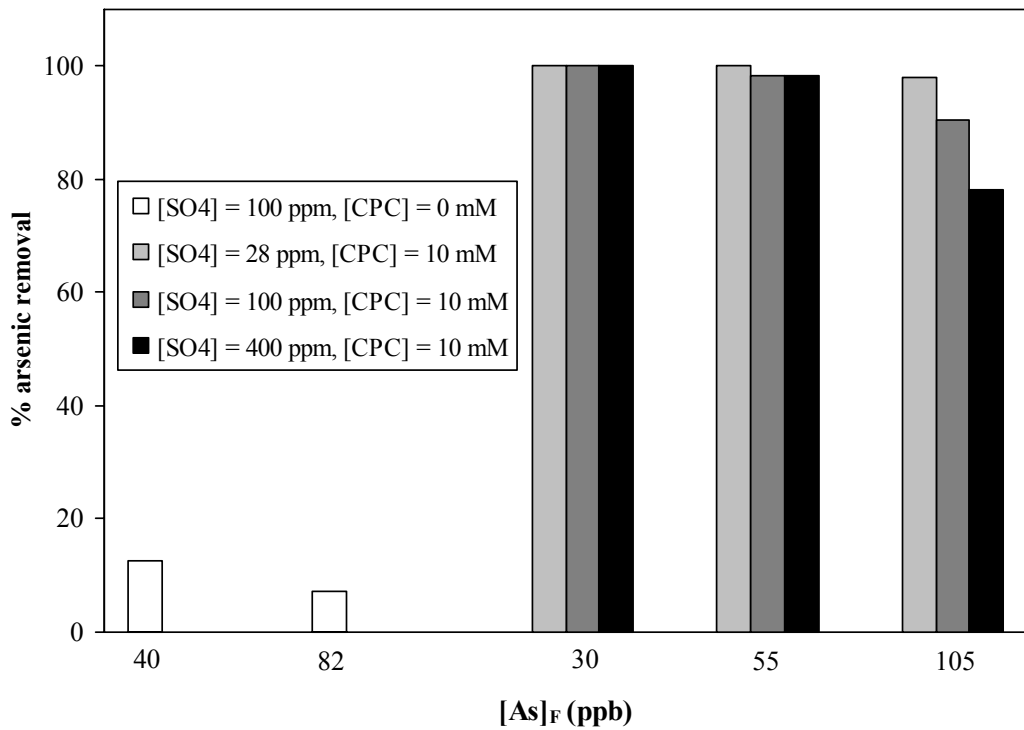


Fig. 4. Effect of SO₄²⁻, feed water arsenic and CPC concentration on As (V) removal.

3.3. CPC Concentration in Permeate Water

CPC molecules present as unimers may pass easily across the membrane barrier since the size of these monomeric units are smaller than the pore diameter of the UF membranes. From both an economical point of view and for the suitability of the final water, the loss of surfactant molecules into the permeate stream is an important parameter. The CPC concentration in the permeate stream is expected to be lower than the CMC (0.90 – 0.95 mM with differing As(V) concentrations) regardless of the CPC concentration in the feed stream [9].

The CPC concentration in the permeate stream was found to be between 0.12 and 0.19 mM (only $17 \pm 3\%$ of the monomers) when a 5 kDa PES membrane is used. This is significantly lower than the CMC. This could be attributed to osmotic pressure effects, the specific pore size distribution, and opposing diffusional flow of the CPC molecules from the membrane surface back into the bulk solution. Overall, only $1.52 \pm 0.33\%$ of the total CPC molecules used were detected in permeate water. These CPC molecules could be further separated using nanofiltration and reused in the feed stream

Conclusion

Using simulated water, the effect of the presence of co-ions (CO_3^{-2} , PO_4^{-3} , SiO_3^{-2} , and SO_4^{-2}) on the removal of arsenic was investigated using cationic CPC micelles and 5 kDa PES membrane. Without the addition of CPC micelles, the PES membrane was found to be ineffective for arsenic removal. In the presence of co-ions, the highest arsenic removal was 25%. However, the addition of CPC micelles significantly increases the arsenic removal efficiency. Regardless of the presence of co-ions, the arsenic concentrations in permeate water were consistently reduced well below (mostly not detected by ICP-MS) the new MCL. The repeatable results obtained for the treatment of simulated water suggest that using ultrafiltration membranes with CPC micelles can be an alternative treatment technology for arsenic removal from contaminated sites.

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