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Removal of arsenic from contaminated water by coagulation followed by polyelectrolyte enhanced ultrafiltration

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ABSTRACT

In this work, removal of arsenic from contaminated water was carried out using coagulation followed by polyelectrolyte enhanced ultrafiltration (PEUF). Ferric chloride and polydiallyldimethylammonium chloride (polyDADMAC) were used as coagulant and cationic polyelectrolyte, respectively. In the first step, effect of coagulant concentration on removal of arsenic was studied and coagulant dosage was optimized. In the second set of study, a combination of low dose of coagulant and polyelectrolyte solution was used for treatment of arsenic contaminated water. The supernatant collected following coagulation was subjected to PEUF so as to remove the residual arsenate ions. Ultrafiltration (UF) was carried out in a tangential flow module fitted with 5 kD polyethersulfone (PES) membrane. During UF, influence of polyelectrolyte dosage and transmembrane pressure (TMP) on permeate flux and arsenic rejection were investigated and optimum values were reported. It was observed that small dose [1 mL PE (polyelectrolyte)/50 mL of sample] could effectively reduce the residual arsenic concentration below permissible maximum contaminant level of 10 ppb. A 99.2% rejection of arsenic was recorded during ultrafiltration at a TMP of 2.5 bar following treatment with 0.234 mM polyelectrolyte solution.

Keywords: Arsenic removal; Polyelectrolyte enhanced ultrafiltration; Coagulant dose; Permeate flux

1. Introduction

In recent times, arsenic in drinking water attracted attention because some of the drinking water resources contain considerable concentrations of arsenic, which causes acute and chronic symptoms in many countries, especially in Bangladesh, China, Mongolia, and Taiwan [1]. Arsenic exposure via drinking water can cause various types of skin lesions, cancers, cardiovascular disease, respiratory, and neurological disorders [2–9].

Within the United States, the Public Health Service first established a maximum permissible concentration of 50 ppb (mg/L) for arsenic in drinking water in 1942. Over the past two decades, there has been re-evaluation of the appropriate maximum contaminant level of arsenic in drinking water [10,11] because

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it is classified as a human carcinogen. In 2001, the United States Environmental Protection Agency (USEPA) implemented a new 10-ppb standard for arsenic in drinking water to be effective in 2006 [12].

The main arsenic species present in natural water are arsenate ions (oxidation state V) and arsenite ions (oxidation state III). Arsenate and arsenite are part of the arsenic acid (H_3AsO_4) and arsenous acid (H_3AsO_3) systems, respectively.

Based on previous studies, As(III) is prevalent in groundwater and 25–60 times more toxic than As(V) [13]. Arsenic occurs in water in different forms depending upon the pH and oxidation potential of water. The effect of redox potential (Eh) and pH on arsenic species has been shown in Fig. 1 [14]. Under oxidizing conditions, $HAsO_4^{2-}$ is dominant at higher pH, H_3AsO_4 will predominate in extremely acidic conditions, and $H_2AsO_4^-$ will predominate at low pH less than pH 6.9.

Various treatment methods have been applied to remove arsenic from drinking water. These methods involve adsorption and co-precipitation using iron and aluminum salts, adsorption on activated alumina, activated carbon and activated bauxite, various membrane processes such as reverse osmosis and nanofiltration [1].

It has been reported in earlier studies that coagulation can effectively remove arsenic from water along with various suspended and dissolved constituents, notably turbidity, color, phosphate, and fluoride [15–17]. Aluminum salts and ferric salts are commonly used for this purpose due to low cost and relative ease of handling. By this process, chemicals transform dissolved arsenic into an insoluble solids which subsequently get precipitated. Dissolved arsenic may also be adsorbed on the solid hydroxide surface due to van der Waals force of attraction and may co-precipitate with other precipitating species. The solids thus formed can be removed through sedimentation and/or filtration. Usually coagulation process is enhanced by adjusting pH and electrolyte concentration to reduce the absolute values of zeta potentials of particles, and by optimizing coagulation kinetics [17]. Recent studies demonstrated that the presence of hardness in water to be treated could favor removal of arsenic, but that some anions, especially phosphate, carbonate, and silicate, may compete with arsenic for the sorption sites, thus interfering with removal of arsenic [18].

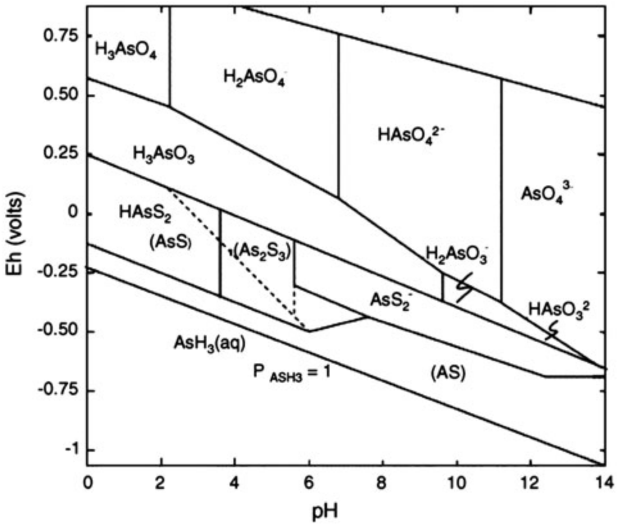

Fig. 1. The Eh–pH diagram for arsenic at 25℃ and 101.3 kPa.

In case of removal of arsenic by adsorption, regeneration of adsorbent would be a difficult task. If coagulation process was solely used for removal of arsenate from solution, a higher concentration of the coagulant would be required to reduce arsenic level below permissible level of 10 ppb and a large quantity of sludge would have been generated.

Among high-pressure membrane processes, reverse osmosis, and nanofiltration (using thin film composite NF membrane) can have over 95% arsenic removal efficiency [1], but it would require very high transmembrane pressure (TMP) as driving force, hence would cause high-energy consumption. So this technology would be difficult to use in developing countries' situation such as low annual income and low electric popularization.

UF, though a low pressure membrane process, solely could not be a viable technique for arsenic removal due to the large pore size of membrane. In PEUF, a cationic polyelectrolyte molecule of very large molecular weight has been added to the solution so that negatively charged arsenate ions could combine with the polyelectrolyte to form macromolecules which would be large enough to be sieved through the pores of the UF membrane.

PEUF has been found to remove multivalent ionic species from aqueous solution when present in low concentration. Polyelectrolytes are water-soluble organic polymers used as coagulant aid for water and wastewater treatment [19]. Flocculation is the action of polymers to form bridges between the larger mass particles or flocs and to bind the particles into large agglomerates or clumps [20]. Generally, the polyelectrolyte is used of opposite charge to the target ions, causing the pollutant ions to bind to the polymer due to electrostatic attraction to form macromolecular complexes. These complexes are retained by the membrane in the retentate stream, while the uncomplexed (unbound) ions pass through the membrane to the permeate stream. In previous studies, PEUF has been applied for the separation of cationic metal ions like Cu^{2+} or Cd^{2+} with anionic polymer [21–23] and anionic ions like chromate (CrO_4^{2-}) with cationic polymer [24–28]. In some recent study, chelating copolymers were synthesized for boron removal via PEUF process [29].

However PEUF, if used alone for removal of arsenic, would require a large quantity of polyelectrolyte to reduce the arsenic level from 100 ppb to the permissible level of 10 ppb and the process would have become uneconomic. Hence in the present study, arsenic removal from contaminated water has been investigated by coagulation followed PEUF using polyDADMAC as the polyelectrolyte.

2. Materials and methods

2.1. Materials

Sodium arsenate salt ($Na_2HAsO_4{\cdot}7H_2O$) (mol. wt. 312) AR grade was purchased from Loba Chemie Pvt. Ltd and was used for preparation of stock solution. Cationic polyelectrolyte, polyDADMAC (average molecular weight 1,00,000) was purchased from Sigma Aldrich as 35% solution in water. Ferric chloride, other chemicals required for arsenic analysis using atomic absorption spectrometry (AAS) such as potassium iodide (99%), L(þ)-ascorbic acid (99.7%), sodium chloride (99.5%), calcium chloride (99%), sodium silicate (97%), magnesium chloride (99%), sodium sulfate (99%), sodium hydrogen carbonate (99.8%), and sodium hydrogen phosphate (99%), sodium metasilicate (97%) were obtained from Merck, India. Deionized and distilled water were used to prepare solutions.

2.2. Membrane module

A tangential flow module, Vivaflow 50, fitted with 5,000 molecular weight cut-off (MWCO) PES membrane having 0.0050 m^2 membrane surface was supplied by Vivascience, A.G. Germany. This type of module causes tangential flow of the feed solution across the membrane and is expected to reduce the concentration polarization phenomenon to a great extent. A variable speed Masterflex peristaltic pump was used to regulate the flow rate of the feed in the module, and thus to vary the TMP. The TMP was read from the reading of a pressure gage attached to the module. The PES membrane was a hydrophilic membrane selected due to its durability, applicability in broad pH range (1–14), and chemical stability. Schematic diagram of cross-flow ultrafiltration module set-up is given in Fig. 2.

2.3. Experimental procedure

The experimental procedure includes coagulation followed by polyDADMAC enhanced ultrafiltration in the cross-flow UF module. In the first step, investigation has been carried out to optimize coagulant ($FeCl_3$) concentration only. As increase in $FeCl_3$ concentration during coagulation causes larger amount of sludge generation, it was intended in the next step to use lower concentration of $FeCl_3$ solution. Hence, the next step of this work consisted of coagulation using 5%

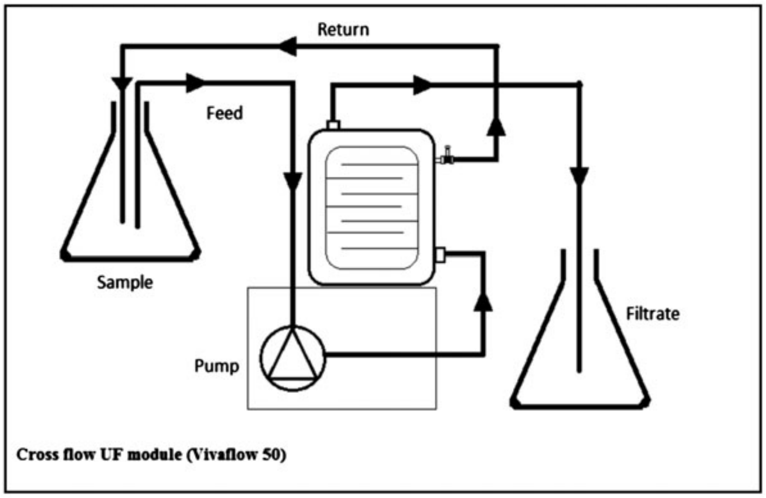

Fig. 2. Ultrafiltration module set-up.

$FeCl_3$ solution followed by PEUF for further removal of arsenic ions.

Effect of pH and PE dosage on arsenic removal have been investigated and reported.

Fifty milliliter of 100 ppb arsenate solutions was kept in nine beakers. Nine different concentrations (5, 7.5, 10, 12.5, 15, 17.5, 20, 22.5, and 25 w/v %) of ferric chloride solutions were prepared using deionized water. Ten milliliter of ferric chloride solutions was added to each beaker containing arsentate solution and pH was increased with 1 N NaOH solution and was magnetically stirred using a magnetic stirrer (REMI) at 150 ± 5 rpm for a period of 15 min. The mixtures were then allowed to settle for another 30 min and then filtered using Whatman (grade 41) filter paper. The supernatant collected in each case was analyzed for residual arsenic concentration.

Second series of experimentation was carried out with an aim to reduce the sludge production. This two-step experiment consisted of coagulation–flocculation using 5% $FeCl_3$ followed by addition of polyelectrolyte (polyDADMAC) solution. To the 50 mL of supernatant thus collected following first step, 1 mL of polyDADMAC solution (35 wt% in water) was added and magnetically stirred for 30 min at 150 ± 5 rpm. To study the effect of PE concentration on arsenic removal during PEUF, similar investigation was carried out by adjusting the ratio of PE solution/feed volume to 2/50 mL and 3/50 mL, respectively. The solution was then ultrafiltered using cross-flow ultrafiltration module until a Volume Concentration factor (VCF) of 2 was achieved. To study the effect of TMP on permeate flux and rejection, ultrafiltration runs were carried out at three different pressures (1.5, 2, and 2.5 bar, respectively) by suitably controlling the regulator fitted with the peristaltic pump. To study the effect of pH on arsenic removal using polyDADMAC addition, the solution pH was adjusted to 5,7, and 9 with the addition of 1 N HCl solution and 1 N NaOH, respectively, prior to the stirring and ultrafiltration. Both the permeate and the retentate thus obtained were then analyzed to detect arsenic concentration using Atomic Absorption Spectrometer (Perkin Elmer). The process flow sheet is given in Fig. 3.

For estimating the performance of PEUF without coagulation, ultafiltration was carried out using 0.078, 0.156 and 0.234 mM PE concentrations at 2.5 bar pressure taking 100 ppb arsenate solution as feed.

2.4. Membrane compaction and water run

Prior to ultrafiltration, the membrane was subjected to compaction for about an hour with ultrapure water at a pressure of 3 bar, higher than the highest operating pressure to prevent any possibility of change of membrane hydraulic resistance during ultrafiltration. Once the water flux becomes steady with no further decrease, it was concluded that full compaction of the membrane had taken place. After compaction, membrane hydraulic resistance (R_m) was determined using Eq. (1) based on water run at different TMPs of 1, 2, and 3 bar.

$$J = \frac{\Delta P}{\mu R_m} \tag{1}$$

where J is the permeate flux, ΔP is the TMP, μ is the solution viscosity, and R_m is the membrane hydraulic resistance.

The membrane hydraulic resistance R_m was calculated. Its value was found to be $(1.07 \pm 0.036) \times 10^{13}\ m^{-1}$.

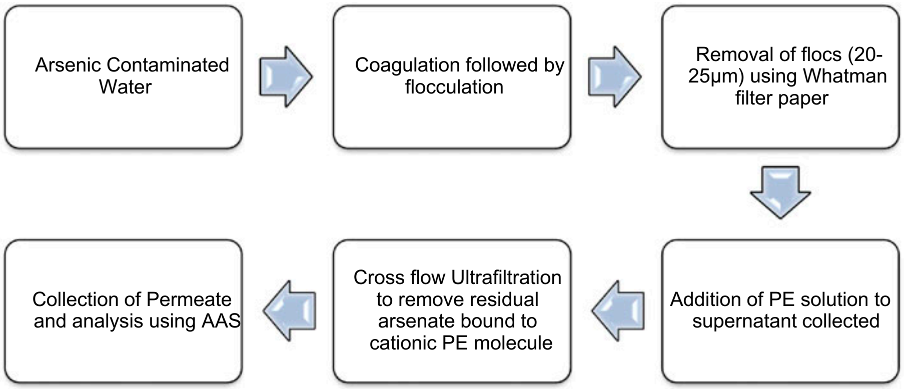

Fig. 3. Process flow sheet.

The term "Rejection" (σ) of solute by membrane is defined by:

$$\sigma = 1 - \frac{C_p}{C_b} \tag{2}$$

where C_p is the solute concentration in permeate and C_b is that of in retentate.

Membrane was washed thoroughly with distilled water after every run with arsenate solution to remove deposited fouling layer, if any; which was followed by water runs to determine the extent of fouling. The variations in water fluxes obtained from such studies were found to be within 2% of initial water flux, thus showing reversible fouling resulting from the proposed separation scheme. The ultrapure deionized water, used in this study was obtained from Arium 611DI ultrapure water system (make: Sartorius A.G., Göttingen, Germany). The feed to this Arium 611DI was taken from usual laboratory distillation unit.

2.5. Analysis

Arsenic concentration was estimated using flow injection hydride generation AAS (FI-HG-AAS) following the *Standard Method for Examination of Water and Wastewater*, number 3114C [30] with a Perkin–Elmer atomic absorption spectrometer fitted with a hydride generator. Before hydride generation, sample solutions containing arsenate were treated with 5% (w/v) of potassium iodide and L(þ)-ascorbic acid, and addition of trace metal-grade HCl so as to reduce arsenic(V) to arsenic(III). Hydride generation was achieved using analytical-grade 0.2% (w/v) sodium borohydride ($NaBH_4$) dissolved in a 0.05% (w/v) sodium hydroxide (NaOH) solution. Using calibration curve, the arsenic concentration of permeate streams was determined.

3. Results and discussion

3.1. Effects of $FeCl_3$ concentration on arsenic removal

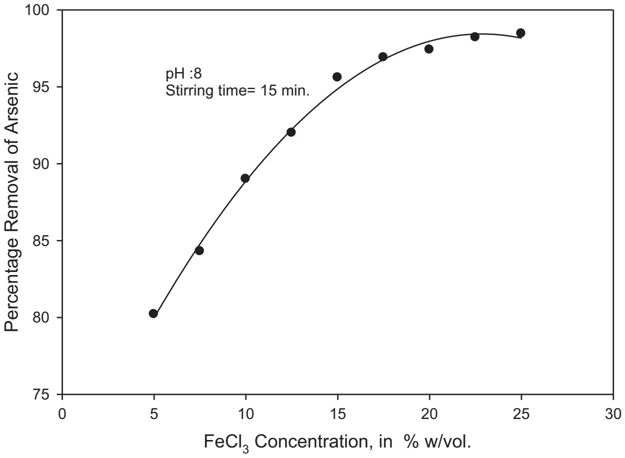

Fig. 4. Effect of $FeCl_3$ concentration on arsenic removal.

Fig. 4 depicts arsenic removal as a function of $FeCl_3$ concentration. It was found that percentage removal of arsenic increased with increase in $FeCl_3$ concentration. Percentage removal of arsenic species increased from almost 80% at 5% $FeCl_3$ concentration to 99% corresponding to 20% $FeCl_3$ concentration. In aqueous solution, ferric chloride hydrolyzed to form a brown acidic solution and the solution became strongly acidic, as was evident from the pH reading. The solution pH was subsequently increased using 1 N NaOH solution. As the pH was increased, precipitation of ferric hydroxide began to appear which formed flocs and co-precipitated with arsenic. It has been observed that percentage removal of arsenic increased with increase in pH in the pH range 5.5–8; beyond pH 8, removal of arsenic got decreased. This trend was observed for different $FeCl_3$ concentrations also. The possible chemical equations related to ferric chloride coagulation were as follows:

$$\begin{aligned} FeCl_3 + 3H_2O \rightarrow {} & Fe(OH)_3 \\ & + HCl \text{ hydrolysis of ferric chloride} \end{aligned}$$

$$H_2A_sO_4^- + Fe(OH)_3 \rightarrow Fe - \text{as complex co-precipitation}$$

During coagulation, removal of arsenic might be taking place by two main mechanisms—adsorption and co-precipitation. During adsorption, soluble arsenic species were incorporated into a growing metal hydroxide phase by occlusion and adsorption. At this pH, As(V) species were negatively charged. Hence, the soluble arsenic got electrostatically attracted and bound to the external positively charged surface of the insoluble metal hydroxides by surface complexation. Since the size of arsenic bearing particles increased, these could easily be separated by ordinary Whatman 41 filter paper, capable of retaining particulates having size more than 20 μm.

As coagulant concentration was increased, more and more arsenate ions got removed from the solution. Beyond 17.5% ferric chloride concentration, the increment in removal percentage of arsenate with increase in coagulant concentration became sluggish.

3.1.1. Effect of pH during coagulation on percentage removal of arsenic

The effect of solution pH on arsenic removal during coagulation process has been depicted in Fig. 4. It may be observed from the figure, that arsenic removal increased from 80% at pH 5.5 to 95.6% at pH 8 when 15% $FeCl_3$ solution was used as coagulant. Beyond pH 8, the removal efficiency was found to decrease. At pH 9 and pH 10, the percentage removal of arsenic decreased to 89.2 and 82%, respectively. Arsenic removal efficiency increased in the pH range 5.5–8, as ferric hydroxide solid is most stable in this range. Due to the solubility of ferric hydroxide at lower pH range, removal efficiency is low (Fig. 5).

3.2. Polyelectrolyte assisted ultrafiltration for arsenic removal

As PEUF is suitable to remove low concentration of ionic species, the supernatant collected following treatment of 100 ppb arsenate solution with 5% $FeCl_3$ solution was used as feed for carrying out PEUF.

The supernatant obtained after coagulation with 5% $FeCl_3$ solution was analyzed and arsenic concentration was found to be 20–21 ppb. To study the effect of a cationic polyelectrolyte on arsenic removal, polyDADMAC, with $[(H_2CCHCH_2)_2N(CH_3)_2Cl]$ as the repeating unit, was used to remove arsenic from water. Due to formation of macromolecular polyelectrolyte–arsenate complexes, rejection of arsenate took place during PEUF. The water-soluble cationic polyelectrolyte, oppositely charged to the target arsenate ions, got attached to the polymer due to electrostatic attraction to form such complexes.

In case of UF, the ability of a membrane to retain a particular solute in a solution depends primarily on the size of solute; it also depends upon surface properties, solute membrane interactions, etc.

The concentrations of arsenic in the permeate stream following UF (up to VCF = 2) at three different TMPs (1.5, 2, and 2.5 bar, respectively) were found to differ slightly. However, as expected, the permeate flux has increased significantly with increase in pressure differential across the membrane. The flux decline phenomenon, i.e. decline of flux with time at a particular TMP which would generally occur (in case of UF and RO) due to concentration polarization or gel layer formation, was not very significant in this case. The variation of flux with time has been given in Fig. 6. It could be seen that 10–12% decline in flux took place after a period of 1.5 h of UF run. The reason could be double folded. As the feed solution was very dilute [containing about 20 ppm As(V) concentration and minute amount of PE], concentration polarization was less significant. Due to the cross-flow, the deposited rejected molecules got swept away with the tangentially flowing feed, thus reducing the polarized layer resistance to a great extent. The flux decline phenomenon during UF has been modeled using resistance-in-series model as given in Eq. (2):

$$J = \frac{\Delta P}{\mu(R_m + R_{cp})} \tag{3}$$

where R_{cp} is the resistance due to concentration polarization.

In each case, following UF, arsenic concentration in the permeate was found to be in the range of 2–10 ppb.

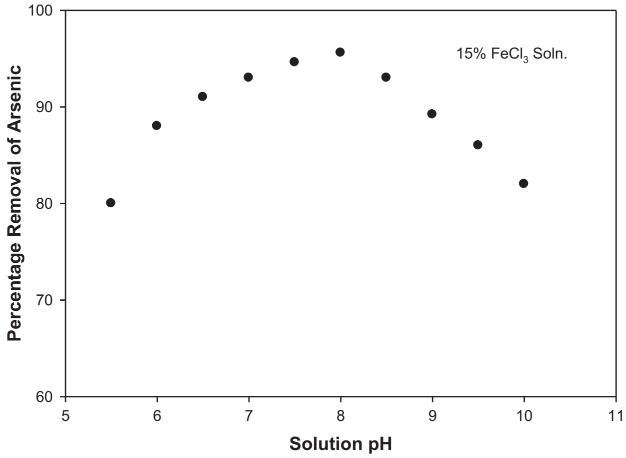

Fig. 5. Effect of pH during coagulation on percentage removal of arsenic.

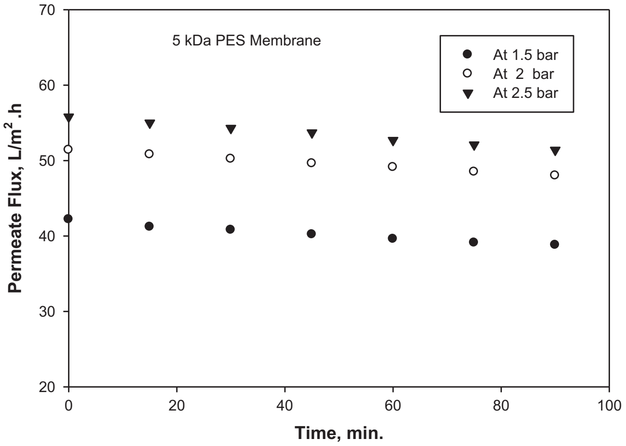

Fig. 6. Flux vs. time during PEUF in cross-flow filtration.

3.2.1. Effect of TMP on permeate flux and rejection

With increase in TMP, steady-state flux increased. The percentage increment of increment steady-state flux first increased with pressure due to increase in driving force. However with further increase in pressure, flux increased but at much slower rate. Gradually, the UF operation was found to be shifted from pressure-controlled region to the mass transfer-controlled region which has been illustrated in Fig. 7.

Arsenic rejection was calculated using Eq. (2). With increase in pressure, average arsenic rejection got increased. Higher TMP caused higher convective flow and accordingly more solute molecules were carried towards the membrane. As a result, more arsenate–polyelectrolyte complex got rejected at the membrane thereby causing a gel/polarized layer to form on the membrane surface which might form a secondary layer on membrane surface offering greater resistance. Due to this, higher rejection of arsenic was observed at higher transmembrane pressures. From the figure, it could be observed that arsenic rejection (at 0.078 mM PE concentration) was increased from 94% at a TMP = 1.5 bar to 98.5% corresponding to a TMP = 2.5 bar.

3.2.2. Effect of PE dosage during PEUF

The effect of PE dosage on arsenate rejection has been studied at three different pH (5, 7, and 9, respectively). From Fig. 8, it may be observed that as the ratio of volume of polyDADMAC solution to arsenate solution was increased from 1 mL PE/50 mL arsenate solution (0.078 mM) to 3 mL PE/50 mL (0.23 mM) arsenate solution, rejection of arsenic increased. Due to increase in number of positively charged sites at higher PE concentration, more and more anions got bound to polyelectrolyte; thus favoring the formation of large sized arsenate–polydiallyldiammonium chloride complex which got retained in the retentate side.

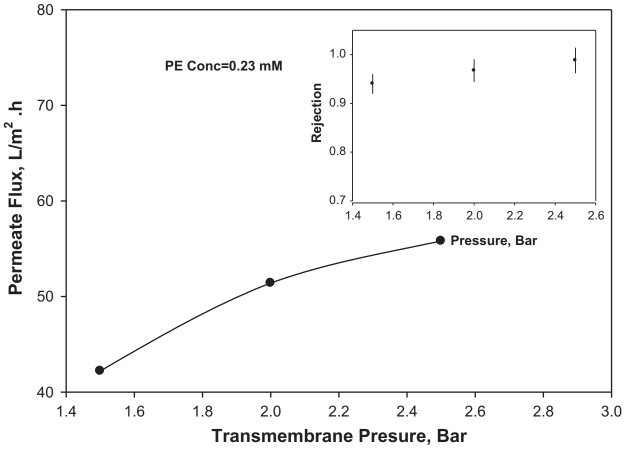

Fig. 7. Effect of pressure on permeate flux and arsenic rejection.

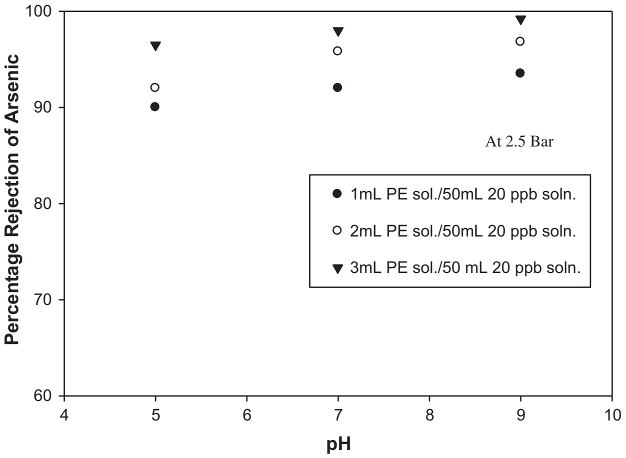

Fig. 8. Effect of solution pH and PE dosage on percent rejection of arsenic.

3.2.3. Effect of pH on arsenic rejection during PEUF

To study the effect of pH on removal of arsenate during PEUF process, to 50 mL of supernatant collected following coagulation using 5% $FeCl_3$ solution, varying dosages of PE solutions were added. In all the cases, the arsenic level were below permissible limit of 10 ppb. Highest percentage removal of arsenic occurred at pH 9 while lowest removal occurred at pH 5. Speciation of arsenic is dependent on pH of the solution. At pH 9, most of pentavalent arsenic exist as $HAsO_4^{2-}$ and AsO_4^{3-} which got more effectively attached to the cationic polyelectrolyte thus facilitating formation of arsenate–polydiallyldiammonium chloride complex which subsequently got rejected in the retentate side. Hence in the permeate following ultrafiltration at a pH 9, lowest arsenic concentration was obtained.

3.3. Comparison of coagulation, PEUF and coagulation followed by PEUF

The comparative performance of three techniques —coagulation, PEUF, and the conjunction of them has been presented in tabular form (Table 1). It has been observed from Table 1, that coagulation process when

Table 1
Comparative performance of coagulation process, PEUF, and combination of coagulation and PEUF

Sl. No.	Treatment processes	Percentage removal of arsenic
1	Coagulation using 5% $FeCl_3$ soln.	80
2	Coagulation using 15% $FeCl_3$ soln. only	95.6
3	Coagulation using 25% $FeCl_3$ soln. only	98.5
4	PEUF using PEUF 0.078 mM solution at 2.5 bar	58.2
5	PEUF using PEUF 0.156 mM solution at 2.5 bar	67.5
6	PEUF using PEUF 0.234 mM solution at 2.5 bar	79.3
7	Coagulation using 5% $FeCl_3$ soln. followed by PEUF (at 2.5 bar) at 0.078 mM PE concentration	98.5
8	Coagulation using 5% $FeCl_3$ soln. followed by PEUF (at 2.5 bar) at 0.234 mM PE concentration	99.2

used singly was effective in reducing arsenic concentration below permissible limit (10 ppb) when at least 15% $FeCl_3$ solution was used. PEUF, when used singly, could only remove 79.3% arsenic (at 0.234 mM concentration) and was not effective in bringing down arsenic concentration below permissible limit. However, a combined treatment consisting of coagulation with 5% $FeCl_3$ solution followed by PEUF using 0.078 mM PE solution was quite effective in lowering arsenic concentration below 10 ppb.

4. Conclusion

The study undertaken in this work was found to be quite effective in reducing arsenic content below permissible limit of 10 ppb. With increase in coagulant $FeCl_3$ dosage, arsenic removal increased. However beyond 17.5% concentration, rate of increase of removal efficiency was not significant. In the second phase of work it was observed, higher polyelectrolyte dosage though caused higher rejection by forming arsenate–polyelectrolyte complex, its impact on flux depressing effect was detrimental. Due to increase in polarized layer resistance at higher concentration of polyDADMAC, the permeate flux reduced. Hence, lower concentration of PE up to 0.156 mM has been suggested. Present investigation also revealed that higher pH favored formation of arsenate complex with cationic polyelectrolyte thereby facilitating highest rejection at solution pH 9. Higher pressure across the membrane was found to have a positive effect on arsenic rejection. A 99.2% rejection of arsenate was recorded at a TMP of 2.5 bar at a PE concentration of 0.234 mM.

Nomenclature

J — flux
ΔP — transmembrane pressure
μ — solution viscosity
R_m — membrane hydraulic resistance
C_p — concentration of solute in permeate
C_b — bulk concentration
R_{cp} — resistance due to concentration polarization

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