



Studies on removal of arsenic using cellulose acetate–zinc oxide nanoparticle mixed matrix membrane

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Abstract

Mixed matrix membranes prepared by varying compositions of Cellulose acetate, acetone and formamide for the synthesis of ultrafiltration/nanofiltration membranes with and without nanoparticles for the removal of arsenic from synthetic solution. Zinc oxide nanoparticles were synthesized by an in situ ultrasonic technique and characterization done using XRD and SEM. In the current study, batch experiments were conducted to characterize the maximum removal efficiency of arsenic by cellulose acetate–ZnO mixed matrix membrane. It was found that 58.77% of arsenic removal was obtained for the feed concentration of 1000 mg/L and pH range 6.8 ± 0.6 . The nanoparticle-embedded membranes show higher removal efficiency, high flux and permeation rate than cellulose acetate membranes without embedded nanoparticles.

Keywords Membrane filtration · Cellulose acetate membrane · ZnO nanoparticles · Arsenic removal · Performance analysis

Introduction

Arsenic is a naturally occurring contaminant, immense concern for the environment and human health because they are highly toxic and present in many contaminated sites, causing an adverse impact on human as well as animal health. Water treatment using membranes is the most popular technology adopted today. Globally, the high levels of arsenic in drinking water are a serious problem [26]. Most of the rural population in India rely on the groundwater sources for drinking purposes, which generally contain underground deposits such as salts and minerals particularly in the West Bengal, India and Bangladesh [8, 10, 34, 38, 39, 47, 63] have stated

that the contamination of groundwater by arsenic [54]. The arsenic (As) occurs in different oxidation states as As(0), As(III), As(V), and As(-III) [9, 13, 14, 24, 57]. However, in oxidizing conditions arsenic appears as oxyanions [12, 46]. In most of the cases, As(III) is 30 times on an average more toxic than As(V) [9, 31]. In most of the countries, Arsenic in groundwater less than 10 µg/L is safe. High concentrations of As are found due to reducing and oxidizing aquifers besides areas affected by industrial, mining and geothermal action [16, 62]. Long-term drinking water exposure leads to lung, skin, kidney and bladder cancer in addition to pigmentation changes, neurological disorders, nausea, hyperkeratosis, muscular weakness and loss of appetite [4, 24, 35].

Conventional treatment methods for the removal of contaminants from groundwater include adsorption, precipitation, electrochemical oxidation and chemical, biological degradation, foam fractionation and membrane technology [1, 50]. Membrane plays a vital role in terms of separation of desired components [3, 48]. As a result, heavy metal separation by membrane filtration attained importance [18, 22, 23]. Development of membranes is a key area of research to synthesize most durable and the high-performance membranes [29, 30]. However, due to the limitations of existing membranes, much research is going on for the synthesis of novel membranes [69, 70] as a thin barrier between miscible fluids [19]. During the separation, a suitable pressure or

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concentration differential is applied across the membrane [20]. Pressure-driven [2] membrane processes can be classified by several criteria like membrane pore size, the pressure exerted on the membrane and charge of the membrane as well as depend on physical properties of the membrane, such as thickness, hydraulic permeability in addition system variables such as filtration time, initial concentration and transmembrane pressure [6, 49, 55, 58]. Nanoparticles have attracted to the amendment of asymmetric mixed matrix membranes with various methods. Using phase inversion processes to fabricate the asymmetric membranes [37], various researchers directly mixed/blended the prepared nanoparticles into the membrane casting solution [5, 7, 15, 53, 65, 67]. The addition of inorganic nanoparticle enhances the hydrophilic and permeability rates of the membranes and shows antimicrobial activity [41, 61, 66]. The incorporated nanoparticles also affect the charge of the membrane, thermal stability, mechanical strength and the membranes showing the antiviral and anti-bacterial properties and also improve the discerning separation, specifically protein separation [27, 45, 59].

In the current study, the inorganic nanoparticles were synthesized using ultrasonication techniques which play in the formation of nanosize particles as reported [59]. Zinc oxide nanoparticles play an important role in antimicrobial, anti-fungal properties and biofouling [17, 21, 51, 56] and nanoparticles are characterized by XRD and SEM [36]. CA/ZnO asymmetric mixed matrix membranes are synthesized using the phase inversion technique [43, 53, 59] and performance analyses of the CA/ZnO membranes like pure water flux, hydraulic permeability, and removal efficiency were analyzed [59].

Materials and methods

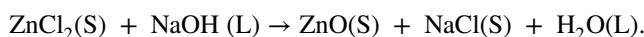
Materials

Cellulose acetate (CA) extra pure with acetyl content ranging from 29 to 45% from lobachemie, Formamide pure, and acetone (purity 99%) was obtained from SRL chemicals which were used as received to prepare the casting solution. Zinc chloride, sodium hydroxide Pure, and PEG 6000

were used to prepare the zinc oxide nanoparticles. Arsenic trioxide extra pure is used to prepare the synthetic solution. Arsenic stock solution was prepared by dissolving 1.320 g in 1 L of deionized water giving a concentration of 1000 mg/L with mild heating.

Synthesis of Zinc oxide nanoparticles

Initially, polyethylene glycol (PEG: molecular weight \approx 6000) and deionized water have been dissolved in 4:1 ratio and allowed on a magnetic stirrer. ZnCl₂ and NaOH [28, 42] with molar ratio 1:1 were dissolved in one deionized water in separate beakers. In the presence of ultrasound, the prepared PEG and ZnCl₂ solutions are allowed to mix for 1 h. NaOH solution was added to the PEG + ZnCl₂ solution mixture drop by drop in the presence of ultrasonication. As a result of heating, crystal nuclei were formed, which then grow as a white precipitate of Zn(OH)₂ was formed. It was filtered and rinsed several times with deionized water until the PEG and NaCl were washed away and the precipitate was dried in a hot air oven at 80 °C for 48 h.



Preparation of cellulose acetate/nanocomposite membranes

The CA asymmetric mixed matrix flat sheet membranes and unmodified CA membranes were prepared in the laboratory by phase inversion solvent evaporation method as described [33, 52] and the casting solution details are listed in Table 1. In the presence of ultrasonication, the zinc oxide nanoparticles were added/dispersed in the casting solution as listed in Table 1. Followed by the casting solutions were kept idle without stirring for 5 min to remove air bubbles. Using a casting/bar coater, the viscous liquid formed has been used to cast the membrane and the casting conditions are given in Table 2.

Characterization of nanoparticles and CA membranes

Nanoparticles have been analyzed using the XRD analyzer (PAN analytical's Xpert Powder) with the parameters: step

Table 1 Composition of casting solution

Membrane casting solution (wt%)	CA1		CA3		CA5	
Cellulose acetate (P)	17	16.80	17	16.80	17	16.80
Acetone (L)	49	48.50	53	52.50	61	60.40
Formamide (L)	34	33.7	30	29.70	22	21.80
ZnO nanoparticle (P)	–	1	–	1	–	1

L liquid, P powder



Table 2 Casting conditions

Temperature of casting solutions (°C)	16–20
Solvent evaporation time (min)	1
Gelation medium	Ice cold deionized water
Gelation time (h)	1

size: 0.016713, time per step: 33.020 s, scan speed: 0.064274, and number of steps: 2633. The size and morphology of the nanoparticles were investigated by particle size analyzer make Malvern and model Zetasizer Nano and observed by scanning electron microscope model Tescan and make Vega 3 LMU. The nanoparticles were suspended in ethanol and deposited on a copper grid base, and allowed ethanol to evaporate. FTIR spectra of CA membrane and mixed matrix ZnO membranes were recorded by the Perkin Elmer spectrophotometer within the range of 450 to 4000 /cm.

Performance studies of membranes

In the current experiment, a lab scale membrane setup containing stainless steel disk membranes had been designed, fabricated (Fig. 6) and used to find the performance analysis of membranes with the effective membrane area in the module which is 28.26 cm².

Pure water flux (PWF)

The PWF measurement was carried out in a batch mode and transmembrane pressure varying from 0.5 to 4 bar. The experiments were performed in an installation consisting of a feed tank, a pump, valve and two manometers. The system pressure and the flow are assured by the pump, and the pressure was adjusted by the valve. The flat plate permeation cells with two detachable parts were separated by a porous plate. PWF has been calculated as follows:

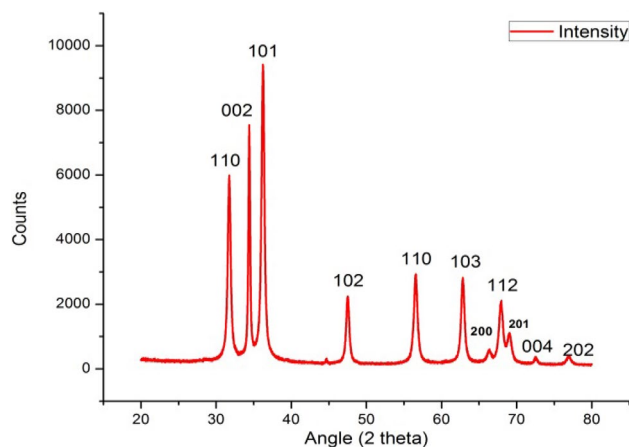
$$J = \frac{V}{A \times \Delta t},$$

where J is the pure water flux (L/M²H¹), V is the amount of permeate collected (L), Δt is the time interval of collection (H) and A is the membrane area (M²).

Hydraulic permeability

The hydraulic permeability of the membrane was obtained from the slope of the straight line between pure water flux and various applied transmembrane pressure. The equation is

$$L_p = \frac{J}{\Delta P},$$

**Fig. 1** XRD characteristic peaks of ZnO nanoparticles

where L_p is the hydraulic permeability (L/M²H¹Bar¹), J is the pure water flux (L/M²H¹), and ΔP is the pressure (bar)

Analysis of arsenic

The concentration of Arsenic in water was analyzed using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), Model-Agilent ICP-OES 700 Series by set the wavelength (187, 197, 198 nm), using the concentration of known standards.

Solute rejection capacity calculation

The efficiency of rejection capability of CA and CA-ZnO nanoparticle mixed matrix membranes, the following rejection equation was used.

$$R = \left(1 - \frac{C_p}{C_f} \right) \times 100\%,$$

where R is the solute rejection, C_p is the solute concentration in permeate and C_f is that in the feed stream.

Results and discussion

Analysis of zinc oxide nanoparticle

Zinc oxide nano-sized particles XRD diffraction patterns are shown in Fig. 1. All the patterns of ZnO nanoparticles were observed as crystal characteristic peaks at 31.76°, 34.40°, 36.26°, 47.58°, 56.51°, 62.77°, 66.36°, 67.94°, 69.10°, 72.53°, and 76.87° corresponding to Chinese white and Zincite, respectively, known as hexagonal wurtzite structure [32, 40, 44]. The wurtzite structure is most stable at ambient



conditions, a good hydrophilic agent which is suitable to modify the membrane.

Figure 2 shows the SEM images of the zinc oxide nanoparticles. The size of the nanoparticles varies from 50 to 500 nm. The result of ZnO using particle size analyzer is shown in Fig. 3. The Z-average particle size of the ZnO is equal to 198.3 nm.

Membrane morphology

To identify the presence of ZnO nanoparticles and the binding of the metal oxide nanoparticle to the polymer matrix were carried out by the FTIR [64]. FTIR spectrum for the membranes CA1, CA3 and CA5 and CA1-ZnO, CA3-ZnO and CA5-ZnO is shown in Fig. 4. A prominent and broad band at 3500–3200 assigned to O–H [60, 68], H-bonded, a strong band at 1742/cm was assigned to the carbonyl C=O stretching [59], band ranging from 3000 to 2850 assigned to C–H [59, 60], a weak band

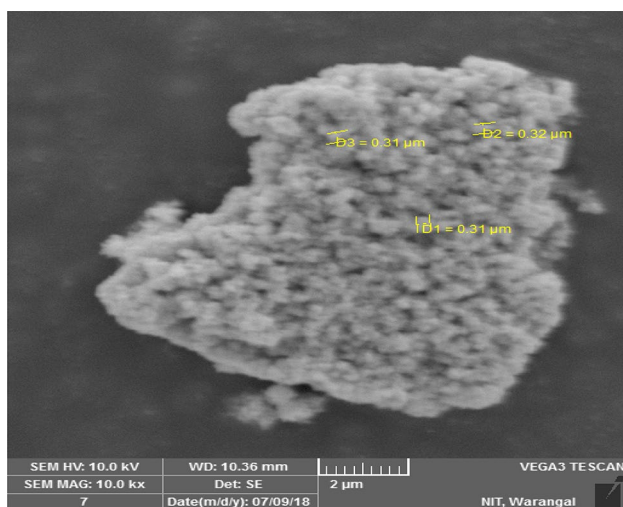
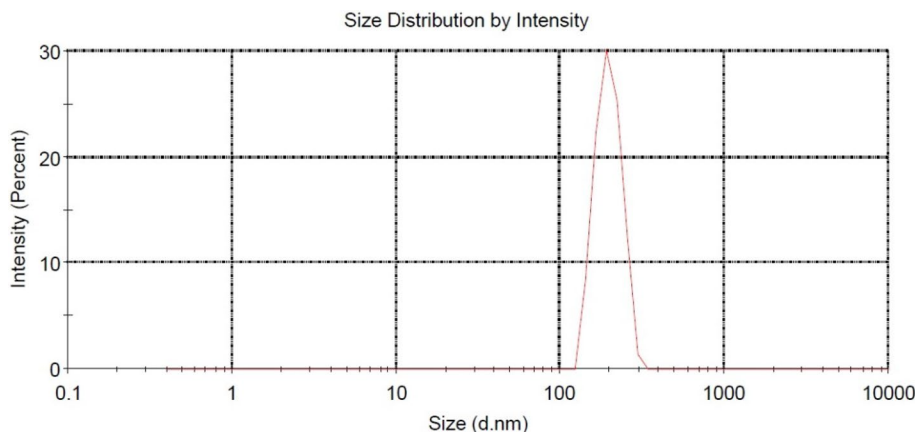


Fig. 2 SEM image of ZnO nanoparticles

Fig. 3 Particle size analysis of ZnO nanoparticles



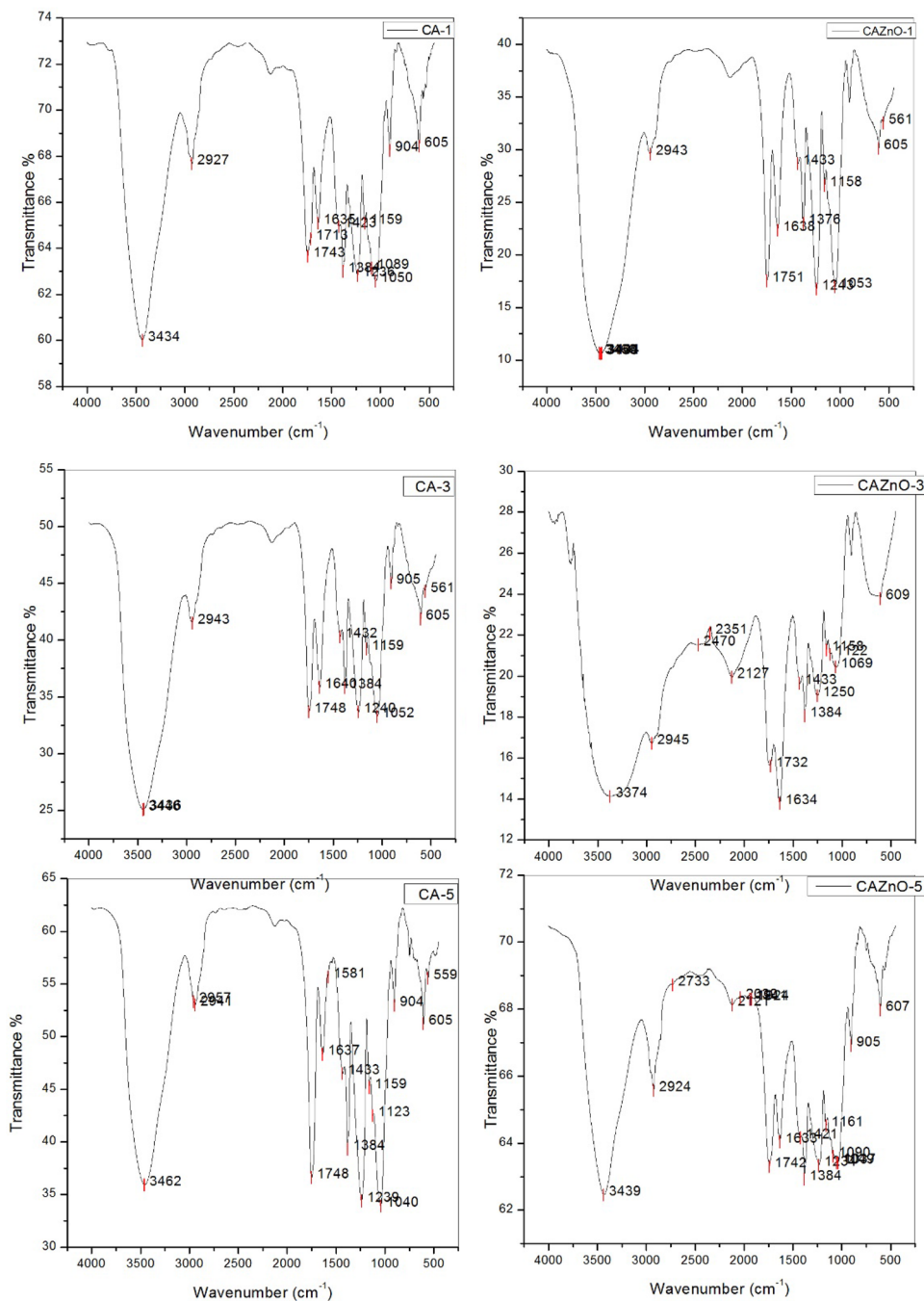
ranging from 2260 to 2100 corresponds to $\text{C}\equiv\text{C}$ stretch. The bending fundamental ($\sim\text{OH}$) for the adsorbed water, which was located at 1636/cm. 1650–1580 assigned to the medium bond of N–H bend, a strong band at 1380–1370 was assigned to CH_3 compound, 1280–1180 range was assigned to strong C–N stretch, 1320–1000 was assigned to the strong C–O stretch, 950–900 [64] the range was assigned to CH_2 out of plane wag. However, the zinc-related band is not observed due to the presence of less quantity. SEM and EDAX were performed for the CA and CA–ZnO membranes shown in Fig. 5A and B, respectively (Fig. 6).

Performance analysis of membranes

Effect of pressure on pure water flux and hydraulic permeability

To determine the pure water flux permeation studies conducted for CA membranes and CA–ZnO membranes are shown in Tables 3, 4 and Figs. 7, 8 and Supplementary Figs. 1–4 and hydraulic permeability is shown in Table 5; deionized water is used for the study. The pure water flux was determined by “weighing the permeate volume (V) collected in a given time (t) and per membrane area (A) as $J = V/(A \times t)$ ”. The hydraulic permeability of the membrane is obtained from “the slope of the straight line between pure water flux and various applied transmembrane pressure”. Transmembrane pressure is ranging from 0.5, 1, 1.5, 2, 3 and 4 bar. The pure flux values of CA membrane with 100 μm and 200 μm thickness were shown against various pressures applied. It has seen that the pure water flux of the membranes increases with increase in the pressure applied. As seen above, pure water flux decreases from CA1 to CA3 membranes and CA1–ZnO to CA5–ZnO. It can be attributed to the lower concentration of swelling agent formamide in CA5 membrane making it less porous. It shows that there was an increase of about 5–25% in the

Fig. 4 FTIR spectrum for the CA and CA–ZnO membranes



flux after adding ZnO nanoparticles. The presence of ZnO nanoparticles shows a positive increase in the hydrophilicity of the membrane making it more porous [11, 25, 32]. The hydraulic permeability of the membranes varied in the order CA1–ZnO > CA1 > CA3–ZnO > CA3 > CA5–ZnO > CA5 for both 100 μm and 200 μm thickness.

In filtration model to overcome the conflicts by the membranes, various pressures were applied to study the removal efficiency of the arsenic from the initial 1000 mg/L concentration solution. In general, flux is directly proportional to

the pressure applied on the system to obtain the maximum arsenic removal without affecting the membrane. In this study, various pressures 0.5, 1, 2, 3 and 4 bars were applied to carry out the study. Till 2 bars of pressure the 100 μm CA membranes are able to sustain the pressure; beyond the 2 bar of pressure, the CA membranes were ruptured. However, the 200 μm CA and CA–ZnO membranes were able to sustain the pressures till 4 bar. Beyond the 4 bar pressure, the only CA–ZnO membranes are able to sustain till 5 bar showing that the incorporation of the ZnO nanoparticles increases the

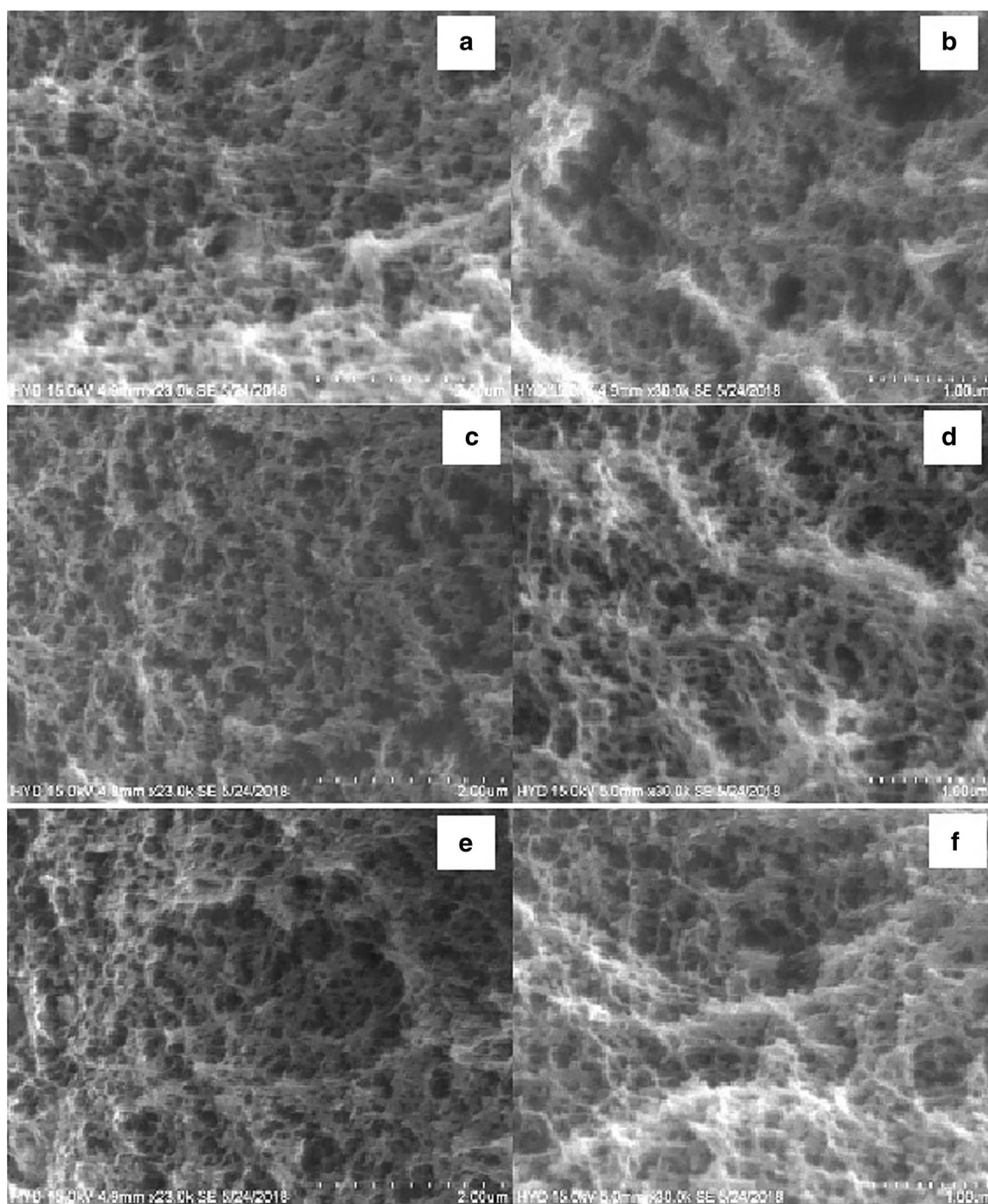


Fig. 5 A SEM for CA and CA–ZnO membranes. Whereas **a**, **c** and **e** are representing CA1, CA3 and CA5; **b**, **d** and **f** are representing CA1–ZnO, CA3–ZnO and CA5–ZnO membranes. **B** EDAX for CA

and CA–ZnO membranes. Whereas **a**, **c** and **e** are representing CA1, CA3 and CA5; **b**, **d** and **f** are representing CA1–ZnO, CA3–ZnO and CA5–ZnO membranes

strength of the membranes. In this context, all the experiments are carried out at 1 bar of pressure to minimize the wear and tear of the CA membranes.

Effect of pH on removal of arsenic

To observe the effect of pH for the removal of arsenic using the cellulose acetate membranes with and without ZnO nanoparticles, the experiments are carried out ranging the pH from 3 to 9. The results showed the good

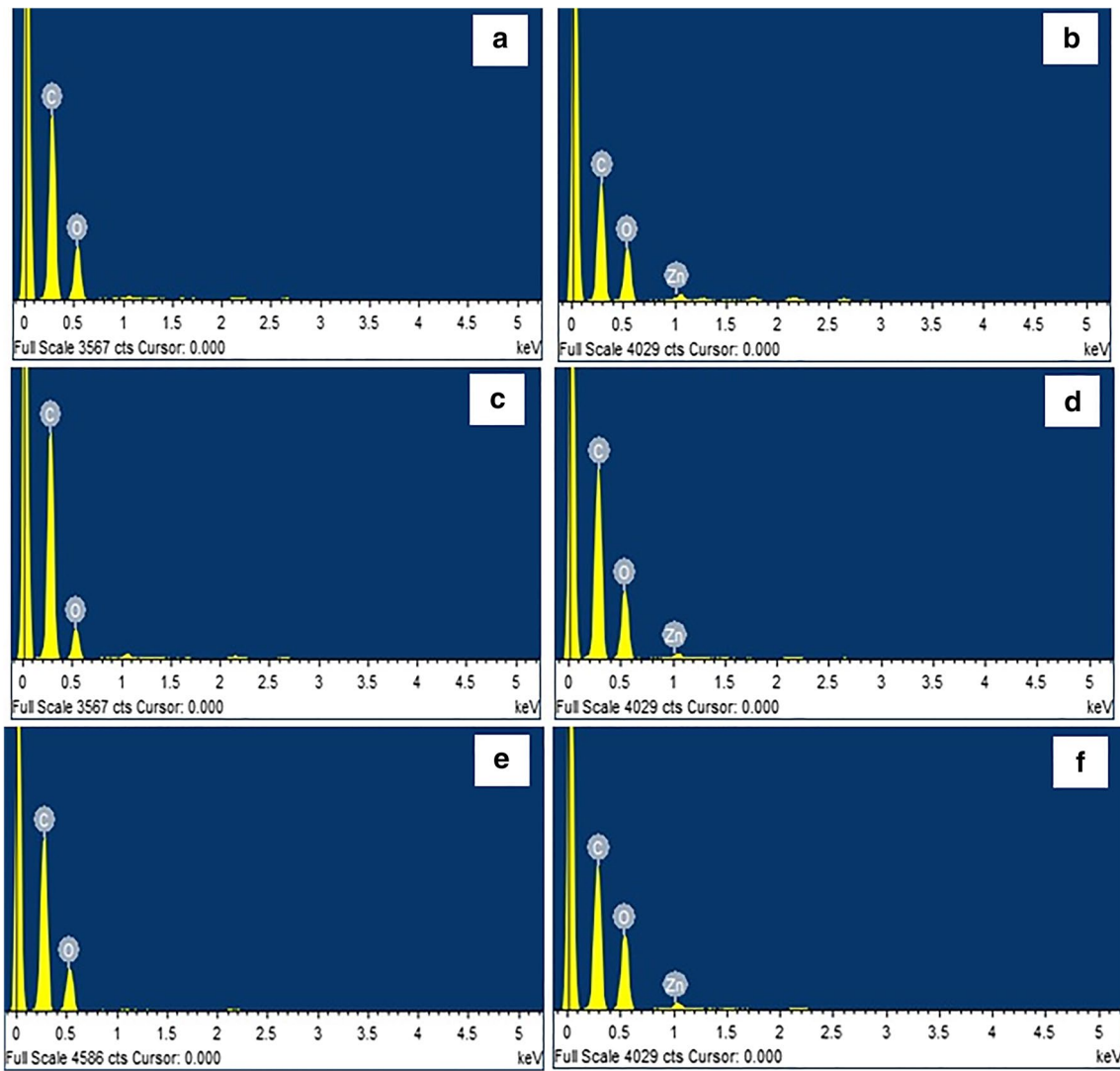


Fig. 5 (continued)

Fig. 6 Schematic representation of experimental setup

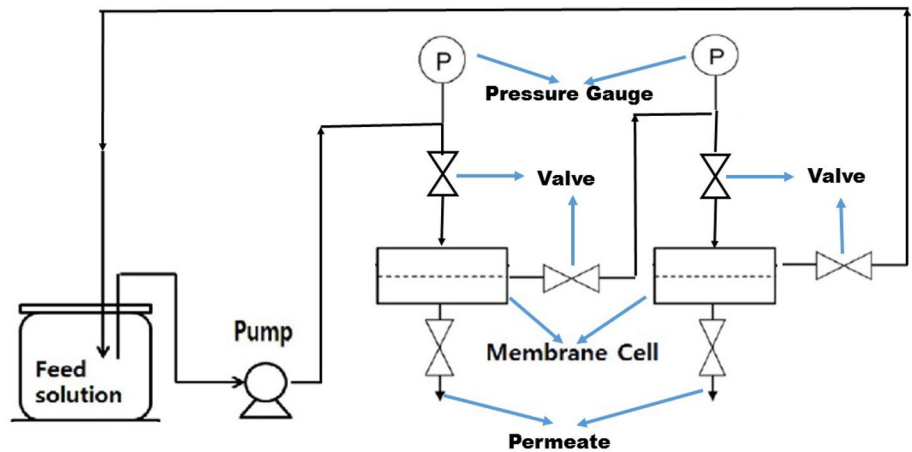


Table 3 Pure water flux for CA 100 μm membranes

Pressure (Bar)	0.5	1	1.5	2	3	4
CA5 flux (L/M ² H ¹)	32.55	43.17	59.07	77.14	107.57	190.65
CA5–ZnO flux (L/M ² H ¹)	34.67	46.70	58.74	84.92	131.63	213.72

Table 4 Pure water flux for CA 200 μm membranes

Pressure (Bar)	0.5	1	1.5	2	3	4
CA1 flux (L/M ² H ¹)	43.87	71.47	96.24	118.28	174.09	255.34
CA3 flux (L/M ² H ¹)	21.23	46.0	53.07	76.77	118.61	178.25
CA5 flux (L/M ² H ¹)	3.24	6.36	19.2	24.4	49.95	63.69
CA1–ZnO flux (L/M ² H ¹)	36.8	66.52	102.61	134.64	206.12	304.31
CA3–ZnO flux (L/M ² H ¹)	18.4	43.87	58.74	85.26	128.89	194.62
CA5–ZnO flux (L/M ² H ¹)	3.95	8.78	19.49	31.89	60.89	79.26

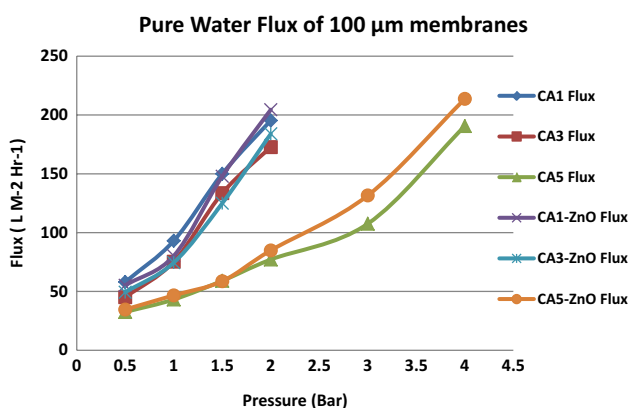


Fig. 7 Pure water flux for CA 100 μm membranes

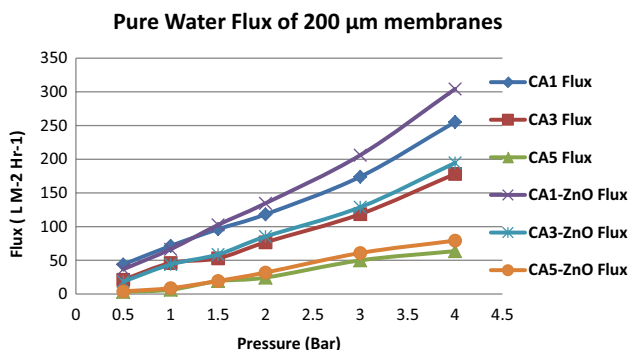


Fig. 8 Pure water flux for CA 200 μm membranes

removal efficiency at the neutral pH 7 only, because ZnO was amphoteric and it dissolves in acidic and basic pH and forms complex like $(Zn(OH)_4)^{2-}$. All the experiments are carried out at pH 7 ± 0.5 .

Table 5 Hydraulic permeability for various membranes

Membrane	Hydraulic permeability 100 μm membrane L_p (L/M ² H ¹ Bar ¹)	Hydraulic permeability 200 μm membrane L_p (L/M ² H ¹ Bar ¹)
CA1–ZnO	103.12	75.496
CA3–ZnO	90.902	49.093
CA5–ZnO	39.519	22.936
CA1	94.162	58.75
CA3	88.194	43.615
CA5	30.768	18.413

Effect of ZnO nanoparticles on removal of arsenic

In the current experiment, different quantities of ZnO nanoparticles were incorporated onto the cellulose acetate membrane to carry out filtration studies for the removal of arsenic. Initially, the 1000 mg/l arsenic stock solution was prepared and used to carry out the arsenic removal studies with CA and CA–ZnO membranes. In CA–ZnO membranes, the ZnO nanoparticles range from 0, 0.25, 0.5, 0.75, 1.0, 1.25, 1.5 g to obtain the final weight of 100 g of casting solution by adjusting the formamide and acetone weights. From the obtained results, it is found that 1 g of ZnO nanoparticles incorporation onto the cellulose acetate (CA5–ZnO) membranes shows the 58.77% of arsenic removal.

Removal efficiency (RE) of arsenic

CA membranes prepared by phase inversion technique were used to study their performance in the removal of arsenic. Using cross flow filtration cell unit, the membrane rejection has been tested under operating pressure of 1 bar, pH 7 ± 0.5 and initial concentration of 1000 mg/L. The membranes were subjected to 4 h of filtration and permeate was

Table 6 Arsenic Removal efficiency of CA membranes

Time (h)	CA1 (200 μm) % RE	CA3 (200 μm) % RE	CA5 (200 μm) % RE	CA1 (100 μm) % RE	CA3 (100 μm) % RE	CA5 (100 μm) % RE
1	30.25	33.45	40.32	31.48	31.84	37.24
2	30.12	33.14	40.25	30.64	31.43	37.12
3	29.16	33.01	38.45	30.01	31.22	36.91
4	27.85	32.17	38.24	29.65	31.01	36.22

Table 7 Arsenic removal efficiency of CA–ZnO membranes

Time (h)	CA1–ZnO (200 μm) % RE	CA3–ZnO (200 μm) % RE	CA5–ZnO (200 μm) % RE	CA1–ZnO (100 μm) % RE	CA3–ZnO (100 μm) % RE	CA5–ZnO (100 μm) % RE
1	51.05	53.32	61.44	45.36	48.88	55.04
2	49.33	51.48	59.32	44.21	47.96	54.17
3	47.24	48.48	57.36	43.54	46.21	53.36
4	45.64	46.24	56.96	42.4	45.18	52.6

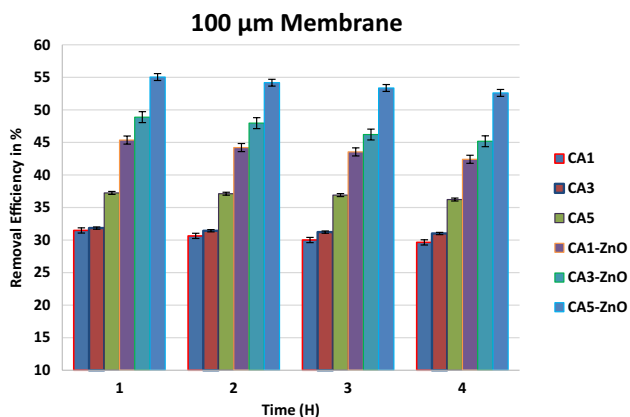


Fig. 9 Arsenic Rejection capacities of 100 μm thickness membranes

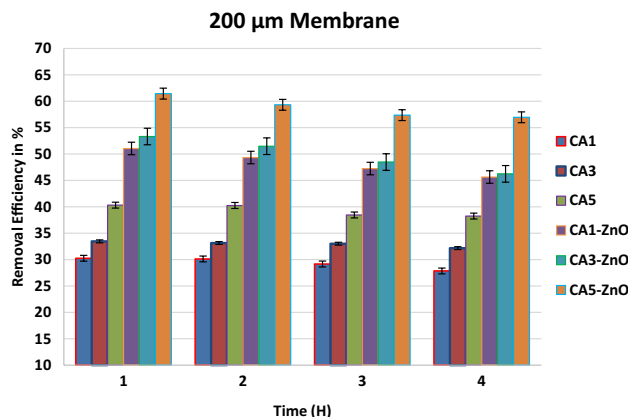


Fig. 10 Arsenic Rejection capacities of 200 μm thickness membranes

collected at every 1 h and the removal efficiency is shown in Tables 6, 7 and Supplementary Tables 1–6.

Figure 9 and Table 6 show the relationship between the % removal efficiency of arsenic with respect to time for different cellulose acetate membranes. From the above figure, the removal efficiency of arsenic from water using CA1 membrane was found to $30 \pm 1\%$ for both 100 μm and 200 μm thickness membrane. The removal efficiency is slightly found to decline after 4 h. For CA3 membrane, the removal efficiency was found to be $33 \pm 1\%$ for 200 μm and $31 \pm 1\%$ for 100 μm thickness. The removal efficiency of arsenic from water using CA5 membrane for 100 μm thickness was found to be $37 \pm 1\%$ and for 200 μm, the removal efficiency was around $40 \pm 1\%$ until 2 h and then decreases to 38.75% at 4 h.

Table 7 and Fig. 10 show the removal efficiency of arsenic by cellulose acetate–ZnO nanoparticle mixed matrix

membrane. For CA1–ZnO membrane, the removal efficiency is found be around $43 \pm 1\%$ for 100 μm and $47 \pm 1\%$ for 200 μm thickness membrane. As compared to CA1 membrane, CA1–ZnO shows higher arsenic removal efficiency. CA3–ZnO membranes arsenic removal efficiency was $48 \pm 1\%$ and $46 \pm 1\%$ for 200 μm and 100 μm, respectively. In the case of CA5–ZnO membranes, the removal efficiency was found to be around $57 \pm 1\%$ for 200 μm and for 100 μm thickness membrane $53 \pm 1\%$. Removal efficiency follows the following trend, CA5–ZnO > CA3–ZnO > CA1–ZnO. Comparing CA membranes and CA–ZnO membranes, the removal efficiency of CA with ZnO nanoparticle membranes was increased.

Membrane charge and pore size play a vital role in separation of ionic species by nanofiltration membrane. The increase in the saturation time and removal efficiency from CA1 < CA3 < CA5 membrane was due to decrease in the

pore size of the membrane and more straining due to the presence of lesser amount of formamide in the preparation of the CA5 membrane. The clogging of the microspaces available till the saturation time further retards the filtration process as explained above.

Conclusion

It can be observed that there was an increase of about 5% to 25% in the flux after adding ZnO nanoparticles. The presence of ZnO nanoparticles shows a positive increase in the hydrophilicity of the membrane making it more porous. As(III) was found to be in the form of the uncharged species H_3AsO_3 in natural waters and removal of As(III) is mainly attributed to steric exclusion than charge effects. Comparing CA membranes and CA–ZnO membranes, the removal efficiency of CA with ZnO nanoparticle membranes was increased.

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