Preparation, characterization and application of cellulose acetate-iron nanoparticles blend ultra filtration membranes

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ABSTRACT

Ultra filtration (UF) membranes are widely applied for micromolecular solutes separation from aqueous streams especially in the field of effluent treatment of chemical process industries. A series of cellulose acetate (CA) blended with iron nanoparticles (modifier) ultrafiltration membranes were prepared by the phase-inversion technique in 100/0, 99.5/0.5, 98.5/1.5 and 97.5/2.5 % polymer blend compositions. The membranes prepared were characterized in terms of compaction, pure water flux, membrane hydraulic resistance, thermo mechanical analysis and water uptake. Surface and cross-sectional morphologies of the membranes were analyzed with scanning electron microscopy (SEM) and atomic force microscopy (AFM). UF characterization studies clearly indicated the better performance of 99.5% CA – 0.5% iron nanoparticles blend membrane in comparison with other synthesized UF membranes. The 99.5% CA – 0.5% iron nanoparticles blend UF membrane was applied to study the rejection of sulphates & organics (in terms of BOD, COD level) for a textile industry effluent. The results indicated almost an equivalent solute rejection percentage (%SR) in comparison to pure CA membranes but with an increased flux rate of separation.

Keywords: Ultra filtration, Cellulose acetate, Iron nanoparticles, Membrane blending, Membrane characterization.

INTRODUCTION

Due to the advantages such as easiness in fabrication as well as operation, compact nature and low energy consumption, membrane separation processes have gained more popularity for industrial separation applications when compared to conventional separation processes. The basic principles of membrane separation and its commercial importance have been extensively reported [1,2]. By blending the base polymer membrane with modifiers, the various properties of the polymeric membrane such as porosity, hydrophilicity, flux and solute rejection can be improved. Polymeric materials and their blends have played an important role in many separation applications such as ultrafiltration (UF), microfiltration (MF), and nanofiltration (NF) [3-8]. For preparation of membranes with better performance, the starting material should be tolerant to wide temperature range and pH range apart from yielding membranes with wider range of pore sizes. Advantages of cellulose acetate (CA) membranes over other membranes like high salt rejection properties and renewable source of raw material has made CA quite popular in ultrafiltration applications. However, pure CA membranes suffer from the limitation of lower fluxes. It has been reported that by blending CA polymer base with other materials, the resulting membrane possessed improved physical properties [9-15]. Hvid et al [9] reported that hydrophilic surfaces are less prone to protein fouling and suggested methods to make the membrane surface more hydrophilic by using suitable modifiers as
surface coatings. CA has been successfully blended with sulphonated Polyether ether ketone (PEEK) polymer. The blend membrane showed increased pore size, higher flux and greater hydraulic resistance [10]. CA–polyurethane blend ultrafiltration membranes with polyvinylpyrrolidone (PVP) as an additive have been prepared and applied to the rejection of proteins, including bovine serum albumin, egg albumin, pepsin and trypsin with the achievement of more than 90% rejection [11,12]. Because of the excellent film-forming properties of CA, preparation of polymeric blend membranes based on CA as base polymer was a potential idea for UF membrane development for commercial applications [13]. With Polyethylene glycol (PEG) as additive, CA and low cyclic dimmer polysulfone blend membrane resulted in increased water flux, porosity and water content [14]. CA with Polyethylene glycol blend membranes have also been investigated [15]. Recently nanoparticles blended polymer composite membrane has become another promising field in membranes development. These membranes exhibit unique mechanical, electrical, optical and thermal properties due to their synergistic and hybrid properties [16]. Such enhancements are induced by the physical presence of the nanoparticles and by the interaction of the polymer with the nanoparticles & the state of dispersion [17-19]. Activities of the modifiers are greatly enhanced when the particle size is in nanometers. It has been reported that Fullerene (C60) nanoparticles filled polysulfone membranes gave higher hydraulic resistance and rejection as compared to the pure polysulfone membranes [20]. In another study, chitosan nanoparticles (CS) and CA were successfully blended. The blend hollow fibre membrane exhibited reasonably good mechanical strength and high porosity [21].

Iron nanoparticles provide good dechlorination to a wide range of chlorinated organics [22], particularly chlorinated olefins and paraffins in addition to excellent thermal stability, thus making iron nanoparticles blend membranes suitable for high temperature biological effluent treatment. Iron nanoparticles are excellent absorbent and its presence in the membrane increases the membrane’s mechanical stability. It has good hydrophilicity and has the ability to enlarge the pores.

In this current study, polymeric blend membranes with CA as base polymer and iron nanoparticles as modifier have been synthesized with varying compositions. The prepared membranes have been characterized in terms of UF characterizations like pure water flux, hydraulic resistance, thermo mechanical analysis, water uptake and morphology characterizations using SEM & AFM. The blend membrane with better UF characterization was then subjected to textile industry effluent treatment to analyze its rejection properties and the results were compared with pure CA membrane’s performance.

**EXPERIMENTAL SECTION**

2.1. Materials
Cellulose acetate (approximately 45% acetyl content) was procured from Mysore Acetate & Chemical Co. Ltd., India. Iron nanoparticles were procured from Reinstine Nanoventures Pvt. Ltd. Dimethyl Formamide (DMF) solvent was obtained from The Precision Scientific Co (Cbe), Trichy, India. Polyethyleneimine (PEI) was obtained from Triveni Interchem Private Limited, Vapi, India. The textile effluent, for the studying the membrane application for solute rejection, was obtained from a Textile mill located at Special Economic Zone (SEZ), Tirrupur, India. Freshly prepared deionized and distilled water was employed for the preparation of gelation bath. All the reagents and materials used in the membrane preparation process were of analytic grade and were used as such in the preparation process.

2.2. Membrane Preparation
Phase Inversion technique is the most versatile and widely used membrane preparation method among the various other membrane preparation methods [25,26]. Phase Inversion is a very flexible technique to obtain membranes with a wide range of morphological varieties in membrane thickness, pore size and pore size distribution [26]. The phase inversion technique is also compatible with most of the materials used for membrane preparation especially with the preparation of CA membranes [13].

The cast solution for membranes preparation was prepared by dissolving required amounts of the CA and iron nanoparticles in DMF solvent as shown in Table 1. About 82.5% of the casting solution was composed of the solvent (DMF) which is the standard solvent concentration level and which was required for complete dissolution of the polymer matrix into the solvent. Calculated amounts of cellulose acetate & iron nanoparticles modifier, in various weight ratios (attributing to a total weight of 4.375 g) were taken and dissolved in DMF solvent in a round bottom flask. The polymeric mixtures were subjected to blending for a period of 2 to 3 hours until homogeneity of
solution was obtained. Increase in levels of modifier concentration led to increased viscosity in the casting solution which made it difficult to attain homogeneity and thus prolonged time consumption for complete blending. During blending, the round bottom flasks were also subjected to slight heating for attaining homogeneity faster. Once the homogeneous cast solutions were formed, the solutions were allowed to cool for an hour to enable the removal of air bubbles in the cast solutions which would otherwise prove detrimental to the membrane structure. The cast solutions were then cast on smooth glass plates with the help of a doctor blade. Prior to casting, separate gelation baths consisting of distilled water (non-solvent) were prepared and the baths were ice-cooled to 10 °C. After 30 sec of solvent evaporation, just after casting, the glass plates along with the polymer film were immersed in the gelation bath. The skin layer formation of the asymmetric membrane took place mainly during the first 30 seconds of the solvent evaporation. After an hour of gelation, the membranes were removed from the gelation bath and thoroughly washed with distilled water to remove the residual solvent from the membrane. The membrane sheets were subsequently stored in distilled water containing 0.1% of formalin solution to prevent microbial growth on the membrane surface.

Table 1: Compositions of cast solutions used for UF membrane preparation

<table>
<thead>
<tr>
<th>Base Polymer CA (in g)</th>
<th>Modifier Iron nanoparticles (in g)</th>
<th>Solvent DMF in ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.353 (99.5% of solute)</td>
<td>0.022 (0.5% of solute)</td>
<td>21.7 (82.5% of solvent)</td>
</tr>
<tr>
<td>4.309 (98.5% of solute)</td>
<td>0.066 (1.5% of solute)</td>
<td>21.7 (82.5% of solvent)</td>
</tr>
<tr>
<td>4.266 (97.5% of solute)</td>
<td>0.109 (2.5% of solute)</td>
<td>21.7 (82.5% of solvent)</td>
</tr>
</tbody>
</table>

2.3. Membrane Characterization

2.3.1. Scanning electron microscopy

Scanning electron microscopy (SEM) (Supra 55-Carl Zeiss, Germany) was used to analyze the morphology of the blend membranes. The membranes were cut into pieces of various sizes and mopped with filter paper. These pieces were immersed in liquid nitrogen for 20–30 s and were frozen. Frozen bits of the membranes were broken and kept in a desiccator. These membrane samples were used for SEM studies. SEM images were taken for top surface and cross-sectional views of the blend membranes.

2.3.2. Atomic Force Microscopy

Atomic Force Microscopy (AFM) (NTEGRA PRIMA-NTMDT, Ireland) was used to analyze small membrane samples taken from the prepared blend membranes. AFM characterization of a membrane is focused on the determination of the surface morphology and pore statistics (pore size, pore size distribution, pore density, surface roughness), correlation of the membrane structure with membrane properties, surface adhesion and membrane fouling. It gives the topographic images of membrane samples in 3D by scanning with a sharp tip over a surface. AFM images were taken for the synthesized blend membranes. Surface measurement parameters such as roughness average ($S_a$), surface skewness ($S_{sk}$) and surface kurtosis ($S_{ku}$) values were obtained to compare the differences in the surface structures of the prepared UF blend membranes.

2.3.3. Pure water flux

The prepared membranes were cut into an effective membrane area of 38.5 cm² for use in the UF stirred batch cell having 450 ml capacity. The compaction of fresh membranes were carried out by loading the thoroughly washed membranes in the UF test cell connected to the pressure reservoir with water and subjected to compaction at a pressure of 414 kPa. The water flux was measured every 10 min until steady state was achieved. During compaction, membrane pores get compacted leading to a gradual decrease in water flux until finally steady state flux is achieved. This is due to polymer molecules getting slightly reorganized, resulting in low volume porosity, increased hydraulic resistance and thus low flux across the membrane.

Membranes after compaction were subjected to pure water flux treatment at transmembrane pressures of 69, 138, 207, 276, 345 and 414 kPa. The fluxes were measured under steady state flow. The permeate flux was determined using the equation:

$$J_p = \frac{Q}{A \Delta T}$$
where, \( J_w \) – permeate flux (lit \( m^2 \) h\(^{-1}\)), \( Q \) – quantity of permeate (lit); \( A \) – membrane area (\( m^2 \)), \( \Delta T \) – sampling time (h)

### 2.3.4. Water uptake

Initially, the weights of the dry CA blend membranes were measured. The membranes were then placed in a water bath for 24 h and the weights of the hydrated wet membranes were measured. The percent water uptake was calculated by the equation:

\[
WC = \frac{(W_w - W_d)}{W_w} \times 100
\]

where \( WC \) – water uptake (%), \( W_w \) and \( W_d \) (g) – wet and dry weight of the CA blend membranes respectively.

### 2.3.5. Hydraulic resistance (\( R_m \))

Hydraulic resistance is a measure of hydraulic pressure withstanding capability of the membranes. To determine membrane resistance (\( R_m \)), the pure water flux of membranes was measured at different transmembrane pressures of 69, 138, 207, 276, 345 and 414 kPa, after compaction. The hydraulic resistance of the membranes was then evaluated from the inverse of the slope for the plot between water flux (\( J_w \)) and transmembrane pressure difference (\( \Delta P \)) for each blend membrane.

### 2.3.6. Thermo mechanical Analysis (TMA)

Thermo mechanical analysis of the synthesized UF blend membranes was carried out using STA 409PC Seiko Instruments Inc. A sample of 5 mg of the prepared membranes was exposed to a constant force of 0.1N and was heated from 50°C to 650°C with a constant heating rate of 10°C/min under an inert (nitrogen) atmosphere. The membrane samples were initially vacuum dried for 4 h at 50°C to remove moisture content before the start of the analysis. The glass transition temperatures for the blend membranes were obtained from the analysis.

### 2.3.7. Solute Rejection

Rejection studies (based on dead end filtration mode) were carried out in a UF stirred batch cell with an internal diameter of 76mm, 450ml capacity with Teflon coated magnetic paddle. The effective membrane surface area was 38.5 cm\(^2\) and applied pressure was 414 kPa. A constant agitation speed of 500 rpm was used for the paddle in order to reduce the concentration polarization effect. The concentration of feed solution was kept constant. Permeate was collected over defined time intervals in graduated tubes and the tube contents were analyzed for BOD, COD and sulphate content. The BOD, COD and sulphate content analysis results were carried out in the Instrumental & Analytical lab of Tamil Nadu Pollution Control Board (TNPCB).

The solute rejection percentage (%SR) was calculated from the concentration of feed and permeates using the equation:

\[
%SR = \left(1 - \frac{C_p}{C_f}\right) \times 100
\]

where, \( C_p \) and \( C_f \) are solute concentrations of permeate and feed, respectively.

### RESULTS AND DISCUSSION

The morphological and the UF characterization results of the newly synthesized series of CA and iron nanoparticles blend membranes were compared with 100 % pure CA membrane.

#### 3.1. Scanning Electron Microscopy

In asymmetric membranes, the skin layer is mainly responsible for the solute rejection and the flux is determined by the voids in the support layer [13]. As evident from the figure 1, the pure CA membrane show less porosity on the skin surface. The support layer of the same shows uneven distribution and less number of voids, due to which the membranes are likely to exhibit very low flux. In the case of CA and iron nanoparticles (0.5%) blend membrane, addition of modifier has lead to an increase in the number of pores and pore size on the skin layer as well as
enhanced pore size in the supporting polymer layer. With more addition of iron nanoparticles (1.5%), pore size was still increased but the voids in the support layer are found to be highly uneven than that of 0.5% iron nanoparticles blend. Thus, the CA blend membrane with 0.5% iron nanoparticles is expected to give better flux and solute rejection.

![Image](1.1)
![Image](1.2)
![Image](1.3)
![Image](1.4)
![Image](1.5)
![Image](1.6)

Figure 1: SEM images of top surface (1.1, 1.3 & 1.5) and cross-section (1.2, 1.4 & 1.6) of Ultra Filtration membranes – a) 100% pure CA, b) CA– Iron nanoparticles (99.5/0.5%), c) CA– Iron nanoparticles (98.5/1.5%)

3.2. Atomic Force Microscopy

In the AFM analysis, as shown in figure 2, the lighter or the brighter regions shows the nodular structures and the darker regions shows the pores & the depressions. An AFM image depicts the roughness of the membrane and is characterized by the peak structures. The increase in roughness results in an increase in fouling which will in turn cause a fall in the flux rates [26]. In the case of 100% pure CA membrane, the average roughness was found to be 95.3257 nm which is quite high. This is denoted by the large number of peaks distributed throughout the membrane. As a result, pure CA membrane tends to foul more, resulting in low flux transport.

With the addition of iron nanoparticles (0.5%) to the cellulose acetate membrane the peaks are found to be comparatively less, showing less roughness and more pores and thus indicating chances of better fluxes than pure CA. But on further increase in the concentration of iron nanoparticles (1.5%) the average roughness is found to
increase from 16.15 nm (for 0.5% iron nanoparticles) to 58.3 nm (for 1.5% iron nanoparticles). Thus, the CA blend membrane with 0.5% iron nanoparticles is projected to have less effect of fouling and increased flux.

SEM and AFM studies of 97.5% CA – 2.5% iron nanoparticles exhibited the same features like that of 98.5% CA – 1.5% iron nanoparticles indicating lower fluxes and increased roughness.

![AFM images](image)

**Table 2: Roughness parameters of the membranes**

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Roughness average ($S_a$) (in nm)</th>
<th>Surface skewness ($S_{sk}$) (in nm)</th>
<th>Surface Kurtosis ($S_{ku}$) (in nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% CA</td>
<td>95.3257</td>
<td>0.255461</td>
<td>1.54911</td>
</tr>
<tr>
<td>CA – Iron nanoparticles (99.5/0.5%)</td>
<td>16.1576</td>
<td>0.236177</td>
<td>2.12289</td>
</tr>
<tr>
<td>CA – Iron nanoparticles (98.5/1.5%)</td>
<td>58.3096</td>
<td>0.289918</td>
<td>0.624748</td>
</tr>
</tbody>
</table>
3.3. Pure Water Flux, Hydraulic resistance and Water content

Results of pure water flux measurements at various transmembrane pressures, hydraulic resistance of the membrane and water uptake of the 100% CA membrane as well as newly prepared CA – iron nanoparticles blend membranes are presented in Table 3.

From the observed results, it can be seen that the addition of more iron nanoparticles to the CA membrane gave a decreasing trend in the pure water flux. However, as seen from figure 3, 99.5% CA – 0.5% iron nanoparticles blend membrane gave higher flux than the pure CA membrane which is consistent with the results of the SEM analysis. This is due to the enhanced number of pores and increased pore size in the skin layer as well as the increased void structures in the support layer which was caused due to the addition of iron nanoparticles.

The hydraulic resistance of the membranes recorded an increasing pattern with increasing concentration of iron nanoparticles which can be attributed to the decreasing porosity in CA – iron nanoparticles blend membranes especially in support layer, with the exception of the 99.5% CA – 0.5% iron nanoparticles blend membrane. The 99.5% CA – 0.5% iron nanoparticles blend membrane shows the least hydraulic resistance because of its high porosity.

Water uptake of the membranes is an indication of the hydrophilicity and flux behavior of membranes. Membranes were thoroughly washed with distilled water before the estimation of water uptake. With the increase in iron nanoparticles concentration in the CA blend membranes, water uptake phenomena depicted an increasing trend which is due to the increased size of the available pores in the skin layer of the asymmetric membrane.

Table 3: Comparison of UF characterization results for the pure and blended membranes

<table>
<thead>
<tr>
<th>Membrane blend Composition</th>
<th>Pure Water flux (lit m(^2) h(^{-1}))</th>
<th>Hydraulic resistance (kPa m(^{-2}) h (^{-1}))</th>
<th>Water Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CA (%)</td>
<td>INP* (%)</td>
<td>69 kPa</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>0</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>99.5</td>
<td>0.5</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>98.5</td>
<td>1.5</td>
<td>0.7</td>
</tr>
<tr>
<td></td>
<td>97.5</td>
<td>2.5</td>
<td>0.4</td>
</tr>
</tbody>
</table>

* INP – Iron Nanoparticles

Figure 3: Pure water flux measurement for the pure and blended membranes
3.5. Thermo mechanical analysis (TMA)

Results of the TMA studies, as shown in figure 4, clearly indicated the increased thermal stability of the blended membranes in comparison to pure CA membrane. The 99.5% CA – 0.5% iron nanoparticles blend membrane exhibited a greater thermal stability than that of pure CA membrane. Further addition of iron nanoparticles (1.5% and 2.5%) to pure CA membrane caused, only a slightly increased thermal stability in comparison with 99.5% CA – 0.5% iron nanoparticles. The glass transition temperatures for the pure CA and the synthesized blend membranes are given in Table 4.

![Figure 4: TMA curves for the pure and blended membranes](image)

Table 4: Glass transition temperatures of the pure and blended membranes measured by TMA

<table>
<thead>
<tr>
<th>Membrane blend Composition</th>
<th>Glass Transition Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CA (%) Iron Nanoparticles (%)</td>
<td>204.3</td>
</tr>
<tr>
<td>100 0</td>
<td>204.3</td>
</tr>
<tr>
<td>99.5 0.5</td>
<td>396.7</td>
</tr>
<tr>
<td>98.5 1.5</td>
<td>410.3</td>
</tr>
<tr>
<td>97.5 2.5</td>
<td>433.4</td>
</tr>
</tbody>
</table>

3.6. Solute Rejection Studies

Analyzing the results of the morphology and the UF characterization, it was clear that the 99.5% CA – 0.5% iron nanoparticles blend membrane was possessing better features in comparison with the other synthesized membranes. Hence the 99.5% CA – 0.5% iron nanoparticles blend membrane was applied to study the solute rejection characteristics of a textile industry effluent obtained from a textile industry located in the SEZ of Tirrupur, India.

The blend membrane was subjected to textile effluent treatment in the ultra filtration set up under a transmembrane pressure of 414 kPa. The results of the solute rejection studies are presented in Table 5a & 5b. Feed, permeate and reject streams were characterized for their Biological oxygen demand (BOD), Chemical oxygen demand (COD) and sulphate content. BOD and COD give the measure the organic contents / pollutants in the respective streams. Solute rejection studies were carried out in the effluent in two ways – i) with and ii) without addition of Poly ethyleneimine (PEI) to the feed effluent. PEI is a chelating agent and is often used to trap heavy metal ions in them [24]. Thus, a small amount of PEI was added to the effluent and then subjected again for solute rejection studies. Table 5a & 5b shows the analysis of the feed, permeate and reject streams without and with the addition of PEI to the feed effluent respectively. The results of the solute rejection studies of the blend membrane were compared with the rejection studies done with 100% pure CA membrane.

As can be seen from Table 5a (without addition of PEI), the CA–iron nanoparticles blend membrane gave a solute rejection of 36.6 % while the pure CA membrane recorded a solution rejection of 53.65 %. The reason for this
difference could be attributed to the smaller pore size and low flux of the pure CA membrane. However, with the addition of PEI to the feed effluent, the CA–iron nanoparticles blend membrane gave an almost on par solute rejection (80.27%) to that of the pure CA membrane (82.14%) as indicated by the results shown in Table 5b. This was mainly due to the role of PEI in agglomerating the micro size metal ion particles and removing them along with the reject stream. Thus 99.5% CA – 0.5% iron nanoparticles blend membrane is expected to give the separation performance close to that pure CA membrane but with a higher flux rate than that of the pure CA membrane for similar applications.

Table 5a: Solute rejection studies for 100% CA and CA – INP (99.5/0.5%) without PEI addition to feed effluent

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>BOD (mg/lit)</th>
<th>COD (mg/lit)</th>
<th>Sulphates (mg/lit)</th>
<th>% Solute Rejection (%SR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Effluent</td>
<td>79</td>
<td>464</td>
<td>2444</td>
<td>-NA-</td>
</tr>
<tr>
<td>100% CA – Reject</td>
<td>206</td>
<td>1104</td>
<td>2162</td>
<td>53.65</td>
</tr>
<tr>
<td>100% CA – Permeate</td>
<td>23</td>
<td>248</td>
<td>128</td>
<td></td>
</tr>
<tr>
<td>CA–INP* (99.5/0.5%) – Reject</td>
<td>115</td>
<td>624</td>
<td>2094</td>
<td>36.6</td>
</tr>
<tr>
<td>CA–INP* (99.5/0.5%) Permeate</td>
<td>37</td>
<td>163</td>
<td>291</td>
<td></td>
</tr>
</tbody>
</table>

* INP – Iron nanoparticles

Table 5b: Solute rejection studies for 100% CA and CA – INP (99.5/0.5%) with PEI addition to feed effluent

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>BOD (mg/lit)</th>
<th>COD (mg/lit)</th>
<th>Sulphates (mg/lit)</th>
<th>% Solute Rejection (%SR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Effluent</td>
<td>264</td>
<td>1184</td>
<td>2840</td>
<td>-NA-</td>
</tr>
<tr>
<td>100% CA – Reject</td>
<td>372</td>
<td>1560</td>
<td>2641</td>
<td>82.14</td>
</tr>
<tr>
<td>100% CA – Permeate</td>
<td>12</td>
<td>99</td>
<td>99</td>
<td></td>
</tr>
<tr>
<td>CA–INP* (99.5/0.5%) – Reject</td>
<td>316</td>
<td>912</td>
<td>2553</td>
<td>80.27</td>
</tr>
<tr>
<td>CA–INP* (99.5/0.5%) Permeate</td>
<td>14</td>
<td>128</td>
<td>132</td>
<td></td>
</tr>
</tbody>
</table>

* INP – Iron nanoparticles

The BOD and COD features of the permeate streams of the 100% pure CA membrane and the blend membrane were comparable indicating the equivalent performance of the new blend membrane with that of pure CA for the textile effluent treatment with the addition of PEI to feed effluent. Similar pattern was absorbed with regard to sulphate content in the permeate streams of the 100% CA membrane and the blend membrane for the textile effluent treatment with and without the addition of PEI.

CONCLUSION

A series of cellulose acetate membrane (CA) blended with iron nanoparticles modifier was prepared (CA/ Iron nanoparticles: 100/0, 99.5/0.5, 98.5/1.5 and 97.5/2.5%). The prepared membranes were characterized by studying the membrane morphology (porosity and roughness), pure water flux, hydraulic resistance, hydrophilicity and thermal stability analysis. The addition of iron nanoparticles to the base CA membrane influences the membrane properties and morphology remarkably. Experiments showed that the CA–iron nanoparticles blend membrane had increased water uptake, increased hydraulic resistance, increased thermal stability and decreased pure water flux, with increasing concentration of iron nanoparticles in the blend membrane. However the 99.5% CA – 0.5% iron nanoparticles blend membrane reported a remarkably better structural and UF characteristic features than the pure CA membrane including increased pure water flux. This was due to the enhanced number of pores and increased pore size in the skin layer as well as the increased void structures in the support layer of the asymmetric blend membrane which was caused due to the addition of iron nanoparticles. The decreased surface roughness of the blend membrane made it less susceptible to fouling than the pure CA membrane. This 99.5% CA – 0.5% iron nanoparticles blend membrane was applied to study the solute rejection characteristics of a textile industry effluent. Results indicated on par percentage solute rejection of the new blend membrane as compared with pure CA membrane when the effluent feed was mixed with Poly ethylenimine (PEI) prior to solute rejection studies. The 99.5% CA – 0.5% iron nanoparticles blend membrane is expected to give separation performance close to that pure CA membrane but with a higher flux rate for other commercial effluent treatment processes.

Acknowledgements

The authors thank Dr. G. Arthanasewaran of National Institute of Technology (NIT) – Tiruchy, Tiruchirappalli for permitting to use NIT’s membrane research laboratory facilities to prepare the blend membranes and characterize
their properties. The authors also wish to thank the office of the Instrumental & Analytical lab of Tamil Nadu Pollution Control Board (TNPCB), Vellore for characterizing the feed, permeate and reject streams of the effluent treatment.

Appendix

List of symbols used:

- % SR - Percentage solute rejection
- \( J_w \) - Pure water flux
- \( Q \) - Permeate quantity
- \( A \) - Membrane area
- \( \Delta T \) - Sampling time
- \( WC \) - Water uptake
- \( W_w \) - Weight of wet membrane
- \( W_d \) - Weight of dry membrane
- \( R_m \) - Membrane hydraulic resistance
- \( \Delta P \) - Transmembrane pressure difference
- \( C_f \) - Solute concentration in the feed
- \( C_p \) - Solute concentration in the permeate
- \( S_a \) - Surface roughness average
- \( S_{sk} \) - Surface skewness
- \( S_{ku} \) - Surface kurtosis
- CS - Chitosan nanoparticles

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