

## Ranking and comparison of draw solutes in a forward osmosis process

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**Abstract.** Forward osmosis (FO) is an emerging technology which can possibly make the desalination process more cost and energy efficient. One of the major factors impeding its growth is the lack of an appropriate draw solute. The present study deals with the identification of potential draw solutes, and rank them. The comparison was carried out among ten draw solutes on the basis of four main parameters namely; water flux, reverse salt diffusion, flux recovery and cost. Each draw solute was given three 24 hour runs; corresponding to three different concentrations; and their flux and reverse salt diffusion values were calculated. A fresh membrane was used every time except for the fourth time which was the flux recovery experiment conducted for the lowest concentration and the change of flux and reverse salt diffusion values from the initial run was noted. The organic solutes inspected were urea and tartaric acid which showed appreciable values in other parameters viz. reverse salt diffusion, flux recovery and cost although they generated a lower flux. They ranked 5th and 8th respectively. All the experimented draw solutes were ranked based on their values corresponding to each of the four main parameters chosen for comparison and Ammonium sulfate was found to be the best draw solute.

**Keywords:** forward osmosis; draw solute; water flux; reverse salt diffusion; and fouling

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### 1. Introduction

Water scarcity is a growing concern of the modern day world. Currently, about one third of the population experiences water scarcity problems. The growing population and economic growth has led to an increased water demand further aggravating the problem. Moreover, temporal and spatial variations of freshwater resources have made their successful management very tiresome. These issues forced people to choose desalination as a possible solution. Conventionally, desalination is carried out through a process termed as reverse osmosis. However, the process is an expensive and energy intensive one. Many research studies have been conducted to overcome the limitations of this process. In the current context of sustainable development and energy conservation, the scope of forward osmosis is not only restricted to desalination, but also for many other engineering applications viz. wastewater treatment, concentrating landfill leachate and liquid food processing. This interest has been spurred by the recent developments in membrane science

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(Cath *et al.* 2006, Al-Zuhairi *et al.* 2015).

Forward osmosis is explained as the movement of water from a region of higher concentration to a region of lower concentration through a semi permeable membrane. Forward osmosis has several advantages over reverse osmosis process such as it doesn't require application of external pressure; rejects a wide range of solutes and the low fouling tendency (Lay *et al.* 2012). Although the forward osmosis process does not require any energy input, the extraction of pure water demands energy. Certain applications using forward osmosis has been creatively modified so as to reduce this energy requirement (Phuntsho *et al.* 2011, Amy *et al.* 2017).

Further, there is a huge scope for research in developing appropriate draw solute and membrane which would result in appreciable flux, low reverse salt diffusion and easy scalability. The present study focus on 1) Identify the parameters of forward osmosis process that are affected by draw solute characteristics, 2) short-listing draw solutes with potential of high efficiency based on a logical criteria, 3) performing lab scale experiments to obtain the values for the identified parameters of different draw solutes and 4) quantitatively evaluating them based on their performance characteristics and rank them accordingly.

## 2. Materials and methods

### 2.1 Experimental setup

Experimental model used in the present study consisted of two Perspex rectangular prisms each of 11×11 cm with their tops open and connected together at the bottom by means of a connector and union. Membrane was placed at the junction of connector and union so as to enable the separation of two solutions based on their concentration gradient.

### 2.2 Membrane

The membrane used is cellulose acetate (CTA) FO membrane. The CTA FO membrane has been named as the best available membrane in FO applications (Cath *et al.* 2006). This is a flat sheet membrane cast on a non-woven support layer which was heat or RF-weldable. The flat sheets were of size 1'×1' and membranes were cut out from the sheet for use in experiments. FO membranes have much lesser thickness than reverse osmosis (RO) (McCutcheon *et al.* 2005).

### 2.3 Chemicals

All the chemicals used in the present study were of analytical grade unless otherwise specified. The following are the chemicals used as draw solutes in the present study: Ammonium sulfate [(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>], Calcium chloride [CaCl<sub>2</sub>], Ferric chloride [FeCl<sub>3</sub>], Magnesium chloride [MgCl<sub>2</sub>], Potassium carbonate [K<sub>2</sub>CO<sub>3</sub>], Potassium iodide [KI], Sodium bromide [NaBr], Sodium nitrate [NaNO<sub>3</sub>], Tartaric acid, and Urea.

### 2.4 Chemical short listing

The chemical short listing started with a list of 190 chemical compounds that were most commonly found. The compounds were of organic as well as inorganic category. All the

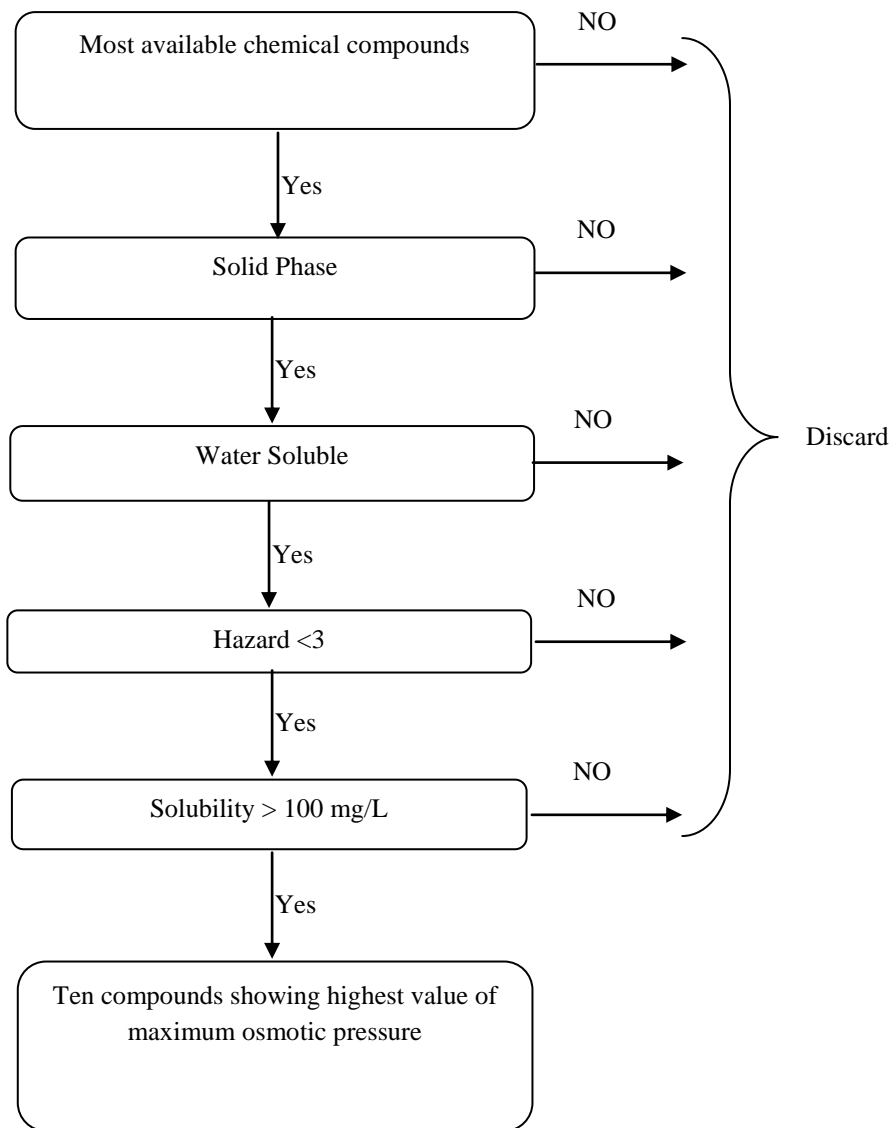


Fig. 1 Flowchart representing the steps in chemical short listing

compounds that are not present as solids at room temperature were excluded from the list since non-solids present problems when it comes to handling and storage. Following this, those chemicals that were not water soluble were discarded from the list since insoluble chemicals do not generate osmotic pressure. The draw solute used may not be completely removed from the system even after separation. Therefore even very low concentrations of highly hazardous chemicals can prove harmful to the users and in case of using the water for potable use, the consequences can be disastrous. Chemicals which show a value greater than 2 (moderate) in any of the categories as given by Hazardous Material Identification System(HMIS) or National Fire Protection Association (NFPA) codes were discarded. The reduced list was subjected to further

shortening by ignoring the chemicals with solubility less than or equal to 100 g/L. Assuming complete dissociation of the chemical species, the maximum osmotic pressure ( $\pi$ ) that can be attained by each of the solute were calculated using Eq. (1). Ten draw solutes which gave the maximum values under this calculation made up the final list of potential draw solutes which were tested for their performance characteristics. The steps involved in the short listing are shown below in the form of a flowchart in Fig. 1.

$$\pi = iMRT \quad (1)$$

where  $i$ , Vant Hoff's factor;  $M$ , Molar concentration;  $R$ , Universal gas constant and  $T$ , Temperature.

### 2.5 Parameter measurement

In order to carry out an FO run, a membrane sample of 7 cm diameter was cut out from the sheet and kept immersed in deionized water for a period of 24 hours. Internal concentration polarization (ICP) is a very important factor that reduces flux in FO process (Chanukya *et al.* 2013, Gray *et al.* 2006). The membrane was so placed that it's active layer was facing the draw side (AL-DS). Since deionized (DI) water was used at the feed side, this leads to a condition of no or minimal ICP and thus a condition of increased flux which will make measurements easy (McCutcheon *et al.* 2006). 1.25 L of required concentration of draw solution was prepared and transferred to one prism of the experimental model (draw side) and 1.75 L of deionized water was taken in the other prism (feed side). A piezometer was connected onto one side of the setup to note the level of liquid. The liquid level right after the draw and feed solutions were transferred was taken as the initial liquid level and the starting time was also noted down. The forward osmosis runs were conducted for 24 hours.

## 3. Results and discussion

### 3.1 Flux measurement

Flux was measured as the volume change of draw solution in unit time. The volume change could be measured from the change in water level. Flux value is given by Eq. (2).

$$J_w = \frac{\Delta V}{A \times t} \quad (2)$$

Where  $J_w$ , water flux;  $\Delta V$ , is the change in volume;  $A$ , area through which water flows measured as the area of connecting pipe; and  $t$ , time required for given volume change taken as 24 hours for all experiments.

Although some intermediate water levels were taken, the intermediate flux values were not of much importance as it is a known fact that the initial flux values are much larger than the subsequent values. The flux was represented as a 24 hour average flux. Molar concentrations were used since they are a more relevant measure as far as forward osmosis is concerned.

The flux values were observed for all the above chemicals at 0.5, 1 and 1.5 M concentrations. Except for urea, none of the other draw solutes showed a linear variation with molar concentration indicating that the forces of concentration polarization (Zhao and Zou 2011). Even though the

Table 1 Flux, reverse salt flux, flux recovery and cost values of the draw solutes

Draw solute	Concentration	Flux values (LMH)	Reverse salt flux (g/m <sup>2</sup> /hr)
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.5 M	9.67	0.39
	1 M	11.52	0.43
	1.5 M	13.98	0.55
CaCl <sub>2</sub>	0.5 M	9.90	0.42
	1 M	10.82	0.17
	1.5 M	12.67	0.16
FeCl <sub>3</sub>	0.5 M	8.52	0.37
	1 M	12.90	0.74
	1.5 M	12.22	1.20
MgCl <sub>2</sub>	0.5 M	10.59	0.27
	1 M	11.98	0.44
	1.5 M	11.03	0.77
K <sub>2</sub> CO <sub>3</sub>	0.5 M	2.36	1.91
	1 M	3.10	15.54
	1.5 M	3.26	20.09
KI	0.5 M	7.33	1.26
	1 M	10.91	2.88
	1.5 M	12.87	6.31
NaBr	0.5 M	7.83	0.41
	1 M	12.41	0.89
	1.5 M	11.89	1.12
NaNO <sub>3</sub>	0.5 M	6.68	0.27
	1 M	10.13	0.57
	1.5 M	10.83	1.30
Tartaric acid	0.5 M	4.61	0.11
	1 M	7.14	0.41
	1.5 M	7.00	0.56
Urea	0.5 M	0.92	0.06
	1 M	2.30	0.25
	1.5 M	3.45	0.09

setup ideally results in no ICP, the effect of external concentration polarization (ECP) cannot be ruled out. Table 1 shows the flux, reverse salt flux, flux recovery and cost values at different molar concentrations of the 10 draw solutes.

There is a general increase in flux with increasing molar concentrations. The highest flux value is shown by (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> at 1.5 M concentration and the lowest value by urea at 0.5 M concentration. However, the maximum value is shown by MgCl<sub>2</sub> followed by CaCl<sub>2</sub> and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> which generates three ions each in solution. The low flux value of the organic solutes can be attributed to their weak dissociation in solution. The value of potassium carbonate is the lowest among the inorganic solutes and is even lower than tartaric acid in spite of the fact that it is capable of generating three ions in solution. The flux value of urea is higher than K<sub>2</sub>CO<sub>3</sub> which

indicates that the increase of flux with molar concentration for  $K_2CO_3$  is very low. The variations observed at the different concentrations can be attributed to the difference in the number of ions released, degree of dissociation and due to CP. Most of the values are in the range of 10 to 14  $L/m^2/h$  (LMH) which is an appreciable flux value.

### 3.2 Reverse salt diffusion measurement

After each FO run, about 250 ml of the feed solution was collected for further analysis. In case of ionic compounds, the analysis for Cations and anions were conducted separately. The shortlisted chemicals contained 8 inorganic salts and 2 organic compounds. While all the 8 inorganic compounds were subjected to cation and anion tests, the organic compounds were tested as a whole. All the tests were conducted according to standard analytical methods (APHA 1999) and the results obtained were expressed in mg/L. The reverse salt flux ( $J_s$ ) value which represents the amount of solute passing through unit membrane area per unit time averaged over a period of 24 hours ( $mg/m^2/hr$ ) is calculated by Eq. (3).

$$J_s = \frac{\text{Total mass of solute on feed side}}{\text{Area of membrane} \times \text{time}} \quad (3)$$

The reverse salt flux values were also obtained for all the chemicals at 0.5M, 1M and 1.5 M concentrations which are shown in Table 1.

While all other draw solutes show comparable values, the draw solutes with 'K' as the cation shows a much higher salt flux value. The lowest values are shown by urea and tartaric acid leading to the conclusion that organic molecules are better rejected by the membrane (Coday *et al.* 2013). The highest salt flux value reached by  $K_2CO_3$  was about  $20 g/m^2/hr$ . Though the values for KI are also high, the highest value attained is only around  $5 g/m^2/hr$  which is much lesser compared to that of  $K_2CO_3$ . These high values shown by the solutes containing the  $K^+$  ion maybe due to the fact

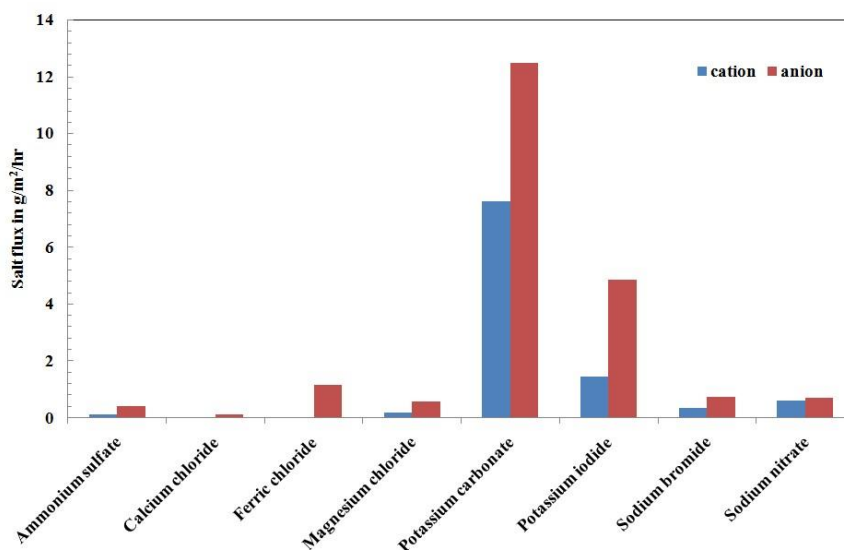


Fig. 2 Cation and anion flux at 1.5 M concentration

that the membrane has low rejection of  $K^+$  ion. Higher reverse salt flux values for  $K^+$  ion is also seen in studies conducted by Achilli *et al.* (2010).

Reverse salt flux values are observed to increase with an increase in the molar concentration with the exception of  $CaCl_2$  and urea. Urea, tartaric acid, calcium chloride and ammonium sulfate show lower reverse salt fluxes.

The separate fluxes of Cations and anions is shown in Fig. 2. Except for the organic solutes i.e., tartaric acid and urea, all the other draw solutes' cation and anion fluxes at different concentrations has been recorded to further understand the reverse salt diffusion process across the CTA membrane.

From Fig. 2, it is seen that the anion flux values are more than the cation flux values irrespective of their hydrated ion sizes. It can also be noted that  $Ca^{2+}$ ,  $Fe^{3+}$  and  $Mg^{2+}$  which have large sized cations show lower cation fluxes. This must have happened in order to maintain electro neutrality (Cath *et al.* 2006, Nayak *et al.* 2011).

### 3.3 Flux recovery

For measuring the flux and reverse salt diffusion, three different concentrations of the draw solutes were tested. Each of these FO runs was conducted with a fresh membrane sample. Flux recovery measurements were carried out for a single concentration i.e., 0.5 M. The membrane sample used in the initial run was reused after immersing it in DI water for more than 48 hrs so as to see how well the system could perform after a simple water backwash. The second run had the same duration of 24 hours, and both flux and reverse salt flux were calculated. Comparison of these values with the initial values with fresh membrane gave the extent of irreversible solids accumulation or damage to membrane (Tang *et al.* 2010, Mi and Elimelech 2010). This measure is useful in determining how frequently the membrane needs to be changed and cleaning needs to be done. This was expressed as the percentage of flux recovery which gives an idea of how much of the initial flux could be recovered by simple immersion in DI water.

$$\text{Percentage flux recovery} = \frac{\text{Flux in second run}}{\text{Initial flux in normal run}} \times 100 \quad (4)$$

From Table 2, it can be seen that the flux recovery values are more than 80 % for all draw solutes except  $CaCl_2$ ,  $FeCl_3$  and  $K_2CO_3$ . This refers to the fact that these chemicals alter the membrane in a permanent way such that its water permeability is affected since this is the only parameter that could possibly vary between the two runs. While  $CaCl_2$  shows a flux recovery around 70 %, the recovery values for  $FeCl_3$  and  $K_2CO_3$  are only around 20%. Lower flux recovery values of  $FeCl_3$  and  $K_2CO_3$  indicates that there might be a permanent alteration or damage caused to the membrane in presence of these solutes and suggests that these solutes are not favorable for use in an FO process.

### 3.4 Cost

The cost of draw solutes was taken as the cost of the chemicals which were available at local supplier i.e., Loba Chemie price list 2014-2015 in US dollar (\$). Cost was calculated as the sum of flux generation cost and replenishment costs. Flux generation cost refers to the cost of draw solute required to produce 1 LMH of flux and Replenishment cost refers to the cost of draw solute required to replenish the draw solute lost due to reverse salt diffusion.

Table 2 Flux recovery values of the draw solutes

Draw Solute	Flux Recovery (%)
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	92.86
CaCl <sub>2</sub>	67.44
FeCl <sub>3</sub>	13.51
MgCl <sub>2</sub>	93.48
K <sub>2</sub> CO <sub>3</sub>	26.96
KI	100.00
NaBr	100.00
NaNO <sub>3</sub>	100.00
Tartaric acid	82.94
Urea	100.00

Flux generation cost for all chemicals was determined considering the average flux per mole values corresponding to all the concentrations. The amount of solute required to generate unit flux was calculated by multiplying the ratio of molarity to water flux ( $M/J_w$  or  $1/J_w$ ) with the molecular weight of the chemical compound. This value when multiplied with the cost per unit weight of the compound gives the flux generation cost.

The replenishment cost was also calculated with the values obtained for 0.5 M concentration of draw solutes. This value was obtained by multiplying the unit cost of the solute with the ratio ( $J_f/J_w$ ). This ratio indicates the amount of solute lost to the feed side per liter of water passing through the membrane (Achilli *et al.* 2010).

A close look at the values in Table 3 shows us that KI has a very high cost per unit weight value which leads to a high flux generation cost i.e., around 3.584 \$ / LMH. Similarly, high reverse salt flux of K<sub>2</sub>CO<sub>3</sub> and the combined effect of cost per unit weight and reverse salt flux results in a high value for K<sub>2</sub>CO<sub>3</sub> and KI i.e., around 0.054 and 0.081 \$ / L. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, CaCl<sub>2</sub>, FeCl<sub>3</sub> and MgCl<sub>2</sub> show consistently low values in both cases and the higher cost per unit weight of CaCl<sub>2</sub> is balanced by a larger flux and lower salt flux.

Table 3 Cost values of the draw solutes

Draw solute	Cost*	
	Flux generation cost (in \$ / LMH)	Replenishment cost (in 10 <sup>-3</sup> \$/ L)
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.057	0.179
CaCl <sub>2</sub>	0.263	0.538
FeCl <sub>3</sub>	0.137	0.717
MgCl <sub>2</sub>	0.108	0.358
K <sub>2</sub> CO <sub>3</sub>	0.565	54.480
KI	3.815	80.824
NaBr	0.200	1.613
NaNO <sub>3</sub>	0.083	0.717
Tartaric acid	0.998	2.509
Urea	0.260	0.538

\*cost calculated in US Dollar (\$) as per the available local market rate for each of the chemical.



### 3.5 Ranking

The ranking of the draw solutes were based on the values of draw solutes corresponding to each of the parameters of concern i.e., flux, reverse salt flux, flux recovery and cost. In order to compare or integrate these values corresponding to different parameters, they need to be normalized. Once the values are normalized, weights can be assigned to the normalized values depending on which parameter is of larger importance. The weighted normalized values can then be used to rank the draw solutes. All the four parameters were given an equal weightage of 1 and the normalizing was done simply by taking a ratio with the minimum or maximum of the observed values.

The flux values were recorded for three different molar concentrations i.e., 0.5 M, 1 M and 1.5 M. The maximum value of flux corresponding to each of these concentrations was noted. This was denoted as  $J_{w,0.5,max}$ ,  $J_{w,1,max}$ , and  $J_{w,1.5,max}$  respectively. All the observed values under 0.5 M concentration was divided by  $J_{w,0.5,max}$  so that the maximum value is 1 and is shown by the observation which is the maximum. This procedure was repeated for 1 M and 1.5 M concentrations. Since the weightage given to the flux parameter is 1, each of the observed values was multiplied with a 1/3 factor and the values at different concentrations were added up so that the solute which shows maximum flux at all concentrations has a value of 1.

The reverse salt flux values were also observed for three different concentrations similar to the flux values but unlike the flux value, minimal value of reverse salt flux is preferred. Therefore the ratio  $J_{s,min}/J_{s,obs}$  was calculated so that larger values gives a lower ratio and vice versa. These values were also multiplied with a 1/3 factor.

The flux recovery values are already normalized and the only calculation done upon them was that they were divided by 100 to bring them down from the percentage value to a ratio value.

The cost values also need to be minimum and the calculations were similar to that employed for reverse salt flux. The cost values were expressed as flux generation cost and replenishment cost. These are not comparable values since they have different units and therefore the calculations on these components of the cost parameter are carried out separately similar to the calculations carried out for different concentrations of flux and reverse salt flux values. The  $Cost_{min}/Cost_{obs}$  values corresponding to flux generation cost and replenishment cost are multiplied with a factor  $\frac{1}{2}$ . The factored values corresponding to each of the draw solutes is added up to obtain the final value.

The values thus obtained for the four identified parameters all on a scale of 1 were then

Table 4 Rank list of the 10 draw solutes

Draw solute	Sum	Rank
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	3.10	1
MgCl <sub>2</sub>	2.69	2
CaCl <sub>2</sub>	2.46	3
NaNO <sub>3</sub>	2.41	4
Urea	2.34	5
NaBr	2.19	6
KI	1.87	7
Tartaric acid	1.76	8
FeCl <sub>3</sub>	1.54	9
K <sub>2</sub> CO <sub>3</sub>	0.57	10

summed. All the draw solutes showed a value corresponding to how near or far they were to the desirable values. The maximum value that could be attained by a draw solute was 4 which would be the draw solute that showed the highest value in all the parameters. Based on these values, the draw solutes were then ranked from 1 to 10 as given in Table 4.

As it is clear from Table 4,  $(\text{NH}_4)_2\text{SO}_4$  ranks higher than the rest of the draw solutes due to its favorable values in flux, cost and flux recovery. The slat flux values are less favorable compared to the others. Potassium carbonate ranks the lowest with undesirable values for all the parameters of concern. The organic solutes urea and tartaric acid ranked 5<sup>th</sup> and 8<sup>th</sup> respectively. Though  $\text{FeCl}_3$  shows a higher flux, the other parameters do not show favorable values putting it in the second last position.

#### 4. Conclusions

Flux values measured indicate that appreciable fluxes ranging to about 14 LMH [ $(\text{NH}_4)_2\text{SO}_4$ ] is achieved with the tested draw solutes. It is observed that organic draw solutes are weaker flux generators than inorganic draw solutes.

Reverse salt flux values indicate that the membrane shows good rejection of all draw solutes except for  $\text{K}_2\text{CO}_3$  and KI. The reverse salt flux values of all draw solutes with the exception of these solutes are below 1.5  $\text{g}/\text{m}^2/\text{hr}$  indicating good rejection. Further it showed that organic solutes showed a much lower salt flux probably owing to their larger sizes in solution.

Flux recovery studies conducted at 0.5M concentration for the solutes showed that Potassium Iodide, Sodium Bromide, Sodium nitrate, urea showed 100% recovery; ammonium sulfate, magnesium chloride and Tartaric acid showed greater than 80% recovery. Whereas  $\text{CaCl}_2$  (67%),  $\text{FeCl}_3$  (13%) and  $\text{K}_2\text{CO}_3$  (27%) showed very low recovery indicating that the membrane has been altered permanently in the presence of these solutes and unsuitable for use in practical applications as this will either require chemical cleaning or replacement of the membrane every 24 hour.

It can be concluded from the study that a draw solute showing the maximum value in all the parameters is not present. However, considering the parameters identified under this study, the best draw solute is  $(\text{NH}_4)_2\text{SO}_4$  which is ranked first with a weighted normalized value of 3.1 for all the four parameters studied, the maximum value attainable being 4.

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