



Hydrophilic Coating Layer by Layer on Polyethersulfone Membranes for Desalination



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Abstract

Water Desalination is becoming an issue for saving the required potable and drinking water due to shrinkage of pure water. Thus, this work deals with antifouling membrane preparation using polymeric compounds of Polyethersulfone/ Polyvinylalcohol (PES/PVA) with nanomaterials to produce antifouling membrane for water desalination. The PES membrane has prepared by phase inversion process followed by different coating ways. The coating with PVA provided the highest hydrophilic surface. The characterization of prepared membranes was carried out using various analysis methods such as scanning electron microscopy (SEM), mechanical properties, porosity and contact angle. The pore size distribution of prepared membranes was determined using the Brunauer-Emmett-Teller (BET) method. The result showed that the best membrane performance has a coating with PVA layer, where rejection reached to 99 %, 95% and 88% as a function of concentration 500, 1500, and 3000 ppm respectively, while the permeate flux reached to 48.4 L/m².h, 32.3 L/m².h and 21.5 L/m².h respectively.

Keywords: Polyethersulfone; Polyvinylalcohol; Phase inversion; Antifouling; Membrane; Desalination

1. Introduction

As a result of a giant requirement of pure water for human, and in different sectors such as agriculture and industry. For that challenge, water desalination is becoming an issue for redeemable the required potable and drinking water due to shrinkage of pure water. The desalination of water by RO membrane is very interesting work particularly in Arab countries such as Egypt. Membrane preparation with high selectivity and high permeability are necessary in the most applications especially in water desalination [1-4]. on by Generally, in membrane technology, the membrane performance (selectivity and permeability) depends on the membrane structure and membrane material [5-8]. Wet phase inversion process is a significant method for membrane preparation. The membrane is prepared by dissolving a suitable polymer such as cellulose acetate, Polyethersulfone

(PES) polyamide (PA), Polyvinylalcohol (PVA), polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG) in a suitable additive as acetone, N-Methyl pyrrolidone (NMP) and formic acid. [9-16]. Approximately the most problems of membranes are membrane fouling which decrease the lifetime of membrane due to the need more excessive chemical treatment and shut down of the desalination unit. According to that, the goal of this work is the preparation of antifouling membranes. The membrane preparation via phase-inversion technique is the well-known process. Peydayesh et al. [17] reported that the optimum parameters for preparation of Polyethersulfone/ polyvinylpyrrolidone (PES/PVP) nanofiltration membrane and noted that addition of 2% PVP to polymer solution, the water contact angle of the membranes reduces from 75 to 64 degree. Furthermore, through the preparation steps, the hydrophilic PVP rises the water–DMAc exchange rate and thus acts as pore forming agent. As observed

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in the SEM images, the membranes fabricated with a higher PVP concentration exhibit a porous structure with wider finger-like macro voids that result in improved the permeate flux and lower rejection of the PES nanofiltration membranes. PVP enhance the hydrophilicity of the membranes, which improve permeate flow [18]. Basri et al. [19] prepared ultrafiltration (UF) membranes from PES/ PVP/ AgNO₃ polymer solution using phase inversion processes, and reported that the membrane formed from 2 wt.% AgNO₃ and PVP of 360,000 Da showed a high concentration of Ag mainly attributable to the high Ag-particle entrapment in the membrane structure. Saljoughi et al. [20] studied the preparation of cellulose acetate/ polyvinylpyrrolidone (CA/PVP) membrane via phase inversion processes and reported that when using 0 to 3 wt.% of PVP concentration in polymer solution results porous membrane with formation of macrovoids. On the other hand, more addition of PVP concentration causes transformation of membrane structure from porous structure to dense because of extinction of macrovoids. Consequently, this observation could be attributed to a slight addition of PVP to polymer solution (up to 3 wt.%) causes instantaneous demixing preponderate, whereas more addition of PVP (up to 6 wt.%), causes delayed demixing occurs. In addition, the PVP is more hydrophilic than CA [21] for that reason blending of PVP/CA increases the membrane hydrophilicity. It also noted that, as the coagulation bath temperature (CBT) increased the macrovoids formation increased on membrane structure. On the other hand, as CBT increased from 0 to 25 °C the pure water flux (PWF) increased, while CBT increased from 25 to 50 °C the PWF is reduced, this is can be ascribed to conspicuous reduction of the membrane hydrophilicity. Primarily, the PWF was associated with membrane structure as well as membrane hydrophilicity as recounted in previous work [22, 23]. K. A. Gebru et al. [24] prepared ultrafiltration membranes from a composition of CA/PVP/TiO₂ nanoparticles via phase inversion process, intended for separation of bovine serum albumin (BSA). It was observed that the addition of PVP/TiO₂ to polymer solution lead to decrease the contact angle and to improve the membrane performance (flux, rejection) compared with the blank CA membrane. The prepared membrane with a composition of (10.5 wt % CA, 4 wt. % PVP, 2 wt. % TiO₂) was demonstrated the highest rejection of BSA (94.3%) and flux recovery ratios (NFR) 91.1%. Subsequently from these pervious works, it is observed that the PVP has used well for doping solution of UF membrane with a good separation. Furthermore, the addition of PVP to polymer solution is possible to increase the nanoparticles-entrapment on the surfaces of prepared membrane, tailoring the antibacterial

properties of fabricated membranes [25]. In previous studies, the asymmetric PES/Mn(acac)₃ blend membranes were prepared by implementing metal-organic compound Mn(acac)₃ in the polymer solution mixture, which led to improving hydrophilicity than bare PES [26]. Abdallah et al. [27] investigated a PES/TiO₂NTs blend membrane by blending PES polymer with titanium dioxide nanotubes to prepare membrane for distillation. The prepared membrane improved the separation of water vapor to 99% with a permeate flux of 18.2 Kg/m².h for 8000 ppm salty water [28]. By blending PES with CA, an improved membrane is produced resulting in a salt rejection of 99% and the permeate flux of 21Kg/m².h for 6000 ppm salty water concentration [29, 30]. Xu et al. [31] prepared asymmetric PVC hollow fiber UF membranes and quoted the effect of addition of PVP and PEG on membrane morphology. they declared that membrane porosity is increased owing to the addition of PEG and PVP as well as the membrane flux is improved due to increasing of membrane porosity. The preparation of nanofiltration PES/PVA membrane [32-35], pervaporation PES/PVA membrane [36-42] is reported also in the previous works. Furthermore, PES/PVA membranes is applied for removal of metal ions from aqueous solutions [43-44] and for gas separation [45], and hollow fiber membrane preparation [46-48]. Jahanshahi et al. [32] fabricated PVA/PES nanofiltration (NF) membranes for the treatment of pulp and paper industry wastewater. By however, Pourjafar et al. [33] modified the preparation of PVA/PES nanofiltration (NF) membranes with adding TiO₂ nanoparticle to improve NF membrane performance. While, Guo et al. [36] prepared PVA/PES composite membrane for pervaporation of ethylene glycol/water mixture. Whereas, Xu et al. [37] prepared PVA/PES pervaporation composite membranes for desalination. The RO membrane is reported and prepared from several polymers such as CA [10], and from copolymer such as CA/PVA [7], and CA/PES [30]. The RO membrane is mainly applied for water desalination. While the preparation of Reverse osmosis (RO) membrane from PES/PVA for water desalination is rarely or not reported in previous works for our knowledge. Thus, in this work the copolymer from PES/ PVA is investigated to benefit from their advantages as reported in our pervious works [10, 30]. Thus, this work aimed to prepare antifouling RO membrane for desalination from PES/PVA.

2. Experimental work

2.1. Materials

Polyethersulfone (Ultrason 6020) was purchased from BASF Germany Company, and N-Methyl

pyrrolidone (NMP) as a solvent was obtained from Sigma Aldrich Company. Triethanolamine (TEA) and sodium dodecyl sulfate (SDS) were purchased from Fluka which were used as additives. Polyvinylalcohol, glutaraldehyde, and acetone were purchased from the Roth India company.

2.2 Membrane preparation

In this work, four RO membranes were prepared by a phase inversion method [10,30], as presented in Table 1. The polymer dope solutions with nano-solution were dissolved in NMP. The nano-solution (NS) was prepared by dispersing 1% titanium dioxide (TiO₂) with 1% triethanolamine (TEA) and 0.5% sodium Deodecylsulfate (SDS) under sonication. 6.5% of NS was mixed with solvent NMP first before addition of Polyethersulfone (PES). Table 1 shows the composition of different prepared polymeric membranes in terms of weight percentages for each ingredient. The dope solution was cast onto nonwoven support fixed on a glass plate and the casting process was carried out using homemade fabricated large-scale casting machine as shown in Fig 1. The thickness of wet membranes was adjusted by casting knife to be 50 µm. Four coating steps were used to make selective layer of RO membranes as shown in tables (1 and 2).

Table 1: Polymeric solution composition for RO membranes

Membrane Symbol	Composition (Weight percentage)			Coating step
	PES %	NS%	NMP%	
R1	24	6.5	69.5	1
R2	23	6.5	70.5	2
R3	23	6.5	70.5	3
R4	25	6	69	4



Fig. 1: Casting machine

Table 2: Coating methods

Coating method	Steps
1	Soaking the membrane in 1% polyethyleneimine in water, then soaking in 0.1% isophthaloyl chloride in hexane, and the final annealing for 15 min at 110°C.
2	Soaking the membrane in 1% polyethyleneimine in water, and then soaking in 0.2% isophthaloyl chloride in hexane. Then the membranes were dried and coated again by 1% polyethyleneimine, 0.1% PVA in water, then soaking in 0.2% isophthaloyl chloride in hexane and the final annealing for 15 min at 115°C.
3	Soaking the membrane in 1% polyethyleneimine in water, and then soaking in 0.2% isophthaloyl chloride in hexane. Then the membranes were dried and coated again by 1% polyethyleneimine, 0.1% PVA in water, then soaking in 0.2% isophthaloyl chloride in hexane and the final annealing for 15 min at 120°C.
4	Soaking the membrane in 1% polyethyleneimine in water, and then soaking in 0.2% isophthaloyl chloride in hexane. Then the membranes were dried and coated again by 1% PVA with 0.3% sodium dodecyl sulfate and 2ml concentrated sulfuric acid in water, then soaking in crosslinking solution of 25% glutaraldehyde and 75% acetone. Then final annealing for 10 min at 100°C.

2.3 Membrane characterization

The characterization of prepared membranes was carried out using various analysis methods such as scanning electron microscopy (SEM), mechanical properties, porosity, and contact angle [13]. The pore size distribution of prepared membranes was determined using the Brunauer-Emmett-Teller (BET) method as in our previous work [9,12]. The evaluations of prepared membranes performances have been investigated using the homemade laboratory desalination testing unit Fig. 2 as explained in previous work [7]. The flux (J) was determined from equation [7,18]:

$$J = Q / (A * t) \quad (1)$$

Where; Q is permeate mass in kg; A is membrane active area in m² and t is time in hour.

The rejection (R) was calculated as [32]:

$$R\% = \frac{(C_f - C_p) \times 100}{C_f} \quad (2)$$

Where, C_f is the concentration of the feed solution and C_p is the concentration of the permeate. The prepared salt solutions of 2000 ppm NaCl solution. Salt solution was prepared using commercial NaCl. The experiments were performed under operating pressure of 25 bar.



Fig. 2: The laboratory desalination testing unit

The prepared membranes were applied using the same apparatus of membrane performance test for long term experiments for 6 months. The membranes surface wash was applied every 8h. The data of permeate flux and salt rejection were recorded. The analysis of tap water from old steel stream contains accumulated rust with low TDS with a value of 278 mg/L in National Research Centre, Egypt.

3. Results and discussions

3.1 Scanning Electron Microscopy (SEM)

The morphology of the prepared membranes was investigated using SEM. Fig. 3. illustrates the SEM cross-section photos of prepared membranes. The R1 membrane displays a thin top dense layer followed by macrovoids/ finger like structure layer, then a porous bottom layer due to non-woven support, the structure of dense top layer depends on the single layer coating (method 1). The R2 membrane has lower polymer content 23% comparing with R1 membrane, but the membrane cross section indicates that the polymeric solution penetrates the support making connection between the support fiber and polymer. However, R2 membrane using coating method 2, which depends on building two coating layers on the membrane surface under annealing in 115°C, which provides a thick dense layer. The R3 membrane like R2 membrane but the difference in the annealing temperature, which was 120°C for R3. The top layer of R3 is thicker than R2. The R4 membrane cross section indicates the highest thick top layer of membrane due to using layer by layer coating method (method 4). However, using high polymer concentration 25% leads to delay in de-mixing between solvent and non-solvent (water in coagulation bath) during membrane formation, which provides thick medium layer over the non-woven support of membrane. Using TiO₂ and SDS in Nano-solution led to form dense top layer before coating, then after coating the thickness of this layer increased. The coating of R4 depends on final crosslinking between PVA and glutaraldehyde which formed transparent gel thick layer on the membrane surface, however, using SDS on this layer exhibits antifouling properties on the membrane surface due to the amphiphilic properties of SDS.

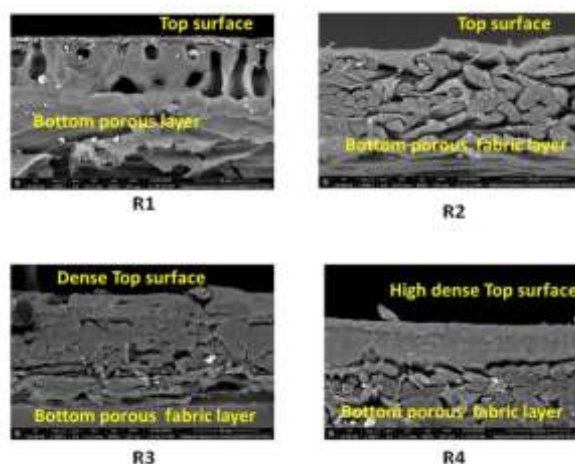


Fig.3:SEM for Prepared Reverse Osmosis Membranes

3.2 Mechanical properties

The RO membranes tensile strength and elongation were measured using the mechanical testing system. Fig. 4 shows the relation between tensile strength and elongation.

The results showed that the maximum tensile strength was (7.7 N/cm²) and the maximum elongation was (19 %) for R4 membrane as shown in Fig.4. Increasing the percentage of polymer to 25 wt% with layer by layer coating increases the mechanical properties. Layer by layer coating provides slightly effect on the tensile strength. However, using nonwoven support is the major parameter for enhancement of mechanical properties of membranes [19-21].

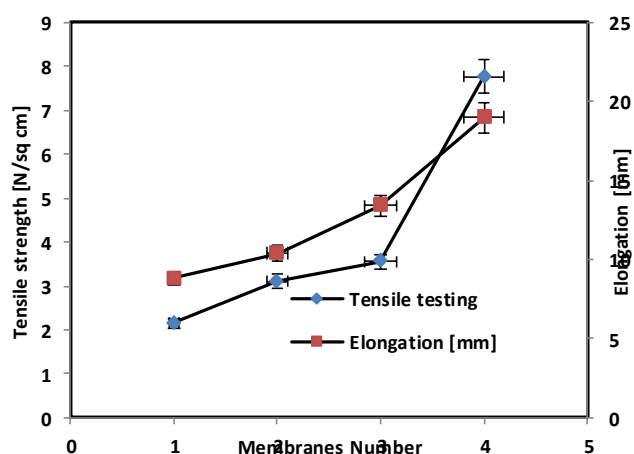


Fig.4 Mechanical Properties for prepared RO membranes

3.3 Porosity measurements and pore size distribution

Table 3 illustrates the overall porosity percentage and average pore size of the prepared membranes. The result indicated that R1 membrane has the highest

porosity (45.7%), while R3 membrane has porosity 4.8 %, R2 membrane has porosity 7.7%, while using layer by layer coating the porosity has reduced to 3.7% for R2 membrane. Using layer by layer coating has reduced the pore size leading to a decrease in the porosity of membranes by inter-polymerization crosslinking reactions [19, 20]. The contact angle measurement indicates that the hydrophilicity of membrane improved depending on the coating layer. The lowest one is at R4 membrane, where the final coating layer has PVA and glutaraldehyde, which make the top surface of membrane super hydrophilic that leads to improve in membrane performance especially in permeate flux.

3.4 Membrane performance test

RO membranes performance test was applied using different synthetic solutions (500-1500 and 3000 ppm) of NaCl. Figs. (5 and 6) show the performance of prepared membranes in terms of the salt rejection percentage and the permeate flux, respectively.

It is clear from Figs. (5, and 6) that the R4 membrane provides the best performance according to rejection percentage and permeate flux. Where, the rejection percentage of salt for R4 membrane reached 99 %, 95% and 88% as a function of concentration 500, 1500, and 3000 ppm, respectively due to the low mean pore size (1.6 nm), which led the improvement in salt rejection. Permeate flux of R4 membrane was the highest one according to the high hydrophilic of the membrane top surface, which was 48.4 L/m².h, 32.3 L/m².h and 21.5 L/m².h as a function of concentration 500, 1500, and 3000 ppm, respectively. The membrane performance as a function of flux and rejection decreased with the increase of feed salt concentration due to increase of osmotic pressure [6,7]. Using triethanol amine in the nano-solution preparation enhances the crosslinking during membrane formation which leads to reduction in void formation. Also, using SDS in the nano-solution which is anionic surfactant because it has anionic head group and hydrocarbon tail, that means it provides amphiphilic properties and exhibits negative charge to the membrane's surface before coating. Using TiO₂ in Nano-solution exhibits dense layer before coating, which leads to a reduction in the permeate flux but according to the hydrophilicity which was enhanced by surface coating the permeate flux was reasonable for all membranes.

Table.3: the porosity and average pore size distribution for prepared membranes

Membrane Type	BET area m ² /g	Total pore volume (cm ³ /g)	Mean pore diameter (nm)	Porosity %	Contact angle
R1	102.6 ± 0.15	2.16 ± 0.15	19.2 ± 0.15	45.7 ± 0.6	66.6 ± 0.8
R2	17.3 ± 0.15	0.364 ± 0.15	3.3 ± 0.15	7.7 ± 0.5	65.1 ± 1.2
R3	10.8 ± 0.25	0.227 ± 0.25	2.06 ± 0.25	4.8 ± 0.7	59 ± 1.4
R4	8.3 ± 0.15	0.175 ± 0.15	1.6 ± 0.5	3.7 ± 1.1	35.3 ± 1.1

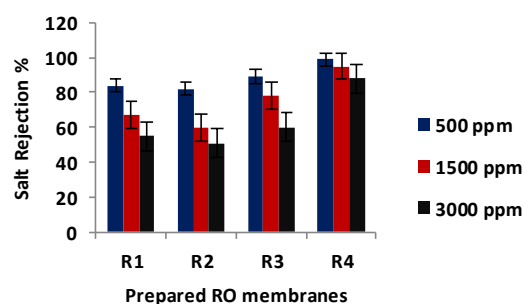


Fig.5: Salt percentage separation as a function of concentration using prepared RO membranes

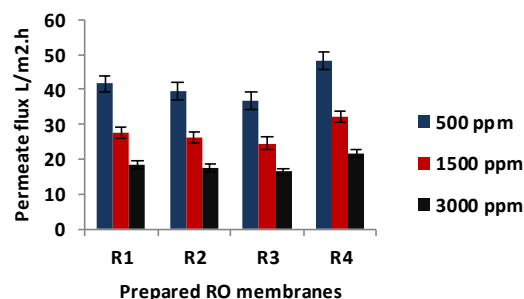


Fig.6: permeate flux as a function of concentration using prepared RO membranes

3.4.1 Long term experiment on the optimum membrane

Long term experiment was carried out using contaminated tap water by rust from old steel stream pipes in National Research Centre, Egypt before changing pipes to PVC pipes. The tap water analysis was illustrated in table (4). R4 membrane was used in this test for 6 months and the average readings were recorded. The membrane was washed every 8 hrs. and reused. Fig.7 indicates the average permeate flux every week from the beginning of the experiment. The results indicate that the flux was approximately fixed for 6 months, which means the R4 membrane was antifouling membrane due to its high hydrophilicity. The hydrophilic properties of titanium dioxide increase the hydrophilicity of the membrane's surface and increase the rate of passing the water through the membrane surface. On the other hand, antifouling properties were enhanced due to the facility of using the same membrane after washing it after using for a long time. Hydrophilicity improves the resistance of membrane to clog. Table (4) indicates TDS reduced from 278 mg/l to 0.97 mg/l which exhibits rejection of 99.7% for most total dissolved salts. Iron and manganese which were considered the reason for the accumulated rust in the wastewater were zeros after treatment by R4.

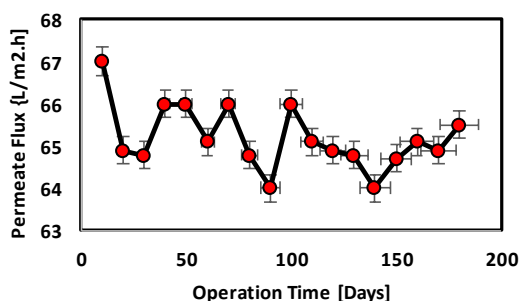


Fig.7. Long term experiment using R4

Table 4: Analysis of water before and after treatment by membrane R4

Parameter	unit	Tap water	After R4	Limits of World Health Organization (WHO) [49]
Chloride	mg/L	45	0.89	<1
Sulphate	mg/L	62	0.76	<1
Total Hardness	mg/L	148	0	< 70
Potassium	mg/L	6.5	0.15	<1
Sodium	mg/L	51	0.98	<1
Magnesium	mg/L	8.5	0	0
Calcium	mg/L	45	0	0
Turbidity	NTU	6.7	0	<1
pH		7.2	6.2	5-8
EC	µs/cm	500	1.5	< 4.7
TDS	mg/L	278	0.97	< 1
COD	mgO ₂ /L	36	0	< 0.5
Fe	mg/L	1.2	0	<0.1
Mn	mg/L	0.01	0	<0.1

Conclusion

The preparations of hydrophilic polyethersulfone membranes using different ways for interfacial polymerization then coating by polyvinylchloride layer were investigated. The main obtained results can be concluded as follow:

- The morphology of the prepared membranes was investigated using SEM, it was found that using the technique of coating layer by layer led to increasing the thickness of dense top layer.
- The coating had not affected mechanical properties, while using the non-woven support enhanced the mechanical properties of membrane.
- The coating layer by layer after preparing membranes by nano-solution enhanced the hydrophilicity of the membrane surface. Where R4 was 35.3° and R1 was 66.6°.
- The performance of R4 membrane was the highest one which was 48.4 L/m².h, 32.3 L/m².h and 21.5 L/m².h and salt rejection was 99 %, 95% and 88% as a function of concentration 500, 1500, and 3000 ppm respectively.
- Long term experiment indicated that R4 membrane is antifouling membrane, which provided good membrane performance for 6 months of operation.

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طبقات تغليف متتالية محبة للماء على أغشية بولي إيثيرسولفون لتحلية المياه

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الملخص:

أصبحت تحلية المياه ضرورية في توفير المياه الصالحة للشرب بسبب النقص الحاد في المياه النقية. ولذلك، هذا العمل يهتم بتحضير الغشاء المضاد للحش (تحمي الأغشية من الانسداد والتلف) باستخدام المركبات البوليميرية من بولي إيثيرسولفون / كحول بولي فينيل (PES / PVA) مع إضافة المواد النانوية لإنتاج غشاء مضاد للحش (مضاد للانسداد والتلف) لتحلية المياه. تم تحضير غشاء بولي إيثيرسولفون من خلال عملية انعكاس الطور متبوعة بطرق طلاء مختلفة. يتم الطلاء للأغشية باستخدام كحول بولي فينيل الذي يجعل سطح الغشاء محب للماء. تم إجراء توصيف الأغشية المحضرة باستخدام طرق تحليل مختلفة مثل الماسح المجهر الإلكتروني (SEM)، والخصائص الميكانيكية، والمسامية وزاوية التلامس. تم تحديد توزيع حجم المسام للأغشية المحضرة باستخدام طريقة Brunauer-Emmett-Teller (BET). تم فحص أداء الأغشية المحضرة باستخدام وحدة تحلية محلية الصنع على نطاق المعمل لاختبار فصل الملح ومعدل السريان للماء الناتج. أظهرت النتائج أن أفضل أداء للغشاء كان الغشاء الذي تم تغطيته بطبقة كحول بولي فينيل، حيث وصلت نسبة فصل الملح إلى ٩٩٪ و ٩٥٪ و ٨٨٪، بينما وصل تدفق الماء الناتج من التحلية إلى ٤٨.٤ لتر/م^٢، ٣٢.٣ لتر/م^٢، ٢١.٥ لتر/م^٢ كدالة للتركيز ٥٠٠ و ١٥٠٠ و ٣٠٠٠ جزء في المليون على التوالي.