



## Prevention of calcium sulfate formation in seawater desalination by ion exchange

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### ABSTRACT

In mechanical vapor compression (MVC) desalination, high operating temperatures (>120°C) allow for high heat transfer coefficients and reduced compressor size, both of which lower total capital costs. To prevent formation of calcium sulfate scale on high-temperature desalination heat exchangers, the inverse solubility of calcium sulfate requires high sulfate removal. To selectively remove sulfate from seawater, a weak-base anion-exchange resin (Relite MG 1/P) was used. In synthetic seawater, the resin shows high sulfate selectivity. Conveniently, the exhausted resin can be regenerated with concentrated acidified (pH 4) blowdown brine from the desalination heat exchanger. Using the same exhaustion and regeneration times, high desalination concentration factors increase chloride concentrations in the blowdown brine, which allows for slower regenerant flow rates thereby increasing regeneration efficiency and sulfate removal. Using a high concentration factor helps regeneration; however, this must be balanced against the greater tendency to form scale in the desalination heat exchanger. With a fixed concentration factor, high flow rates lower the regeneration efficiency and the extent of sulfate removal. In contrast, low flow rates require larger equipment and more resin, which increases capital costs. For a desalination plant with a production capacity of 1 million gallons per day (MGD) and a concentration factor of 2, the cost of sulfate removal – including all operating and capital costs – varies from \$0.246 to \$0.356/thous gallon as the maximum brine temperature changes from 140 to 180°C.

**Keywords:** Calcium sulfate; Ion exchange; Mechanical vapor compression (MVC); Scale; Seawater desalination; Sulfate removal; Weak-base anion exchange resin

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### 1. Introduction

As available fresh water resources decline and demand for potable water grows, economical seawater desalination is increasingly important. The most common desalination techniques include membranes

(reverse osmosis, RO; electrodialysis, ED) and evaporation (multi-stage flash, MSF; multi-effect distillation, MED; mechanical vapor compression, MVC). Despite the rapid deployment of energy-efficient membrane techniques, thermal desalination has the following advantages: minimal pretreatment, flexible salinity in feed water, and high-quality produced water. MSF is more practical when the water resource has high

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salinity and energy costs are not very high, such as in the Middle East. To make thermal desalination energy competitive, a high-temperature MVC was developed with a high-efficiency engine, a novel sheet-shell heat exchanger, and a gerotor compressor [1]. An important factor limiting the application of conventional low-temperature MVC ( $<80^{\circ}\text{C}$ ) is the high capital cost of the compressor, which must be large enough to accommodate low-density vapor. Increasing the MVC temperature increases vapor density, thus shrinking the compressor size and lowering the capital cost. In addition, heat transfer coefficients are much higher at elevated temperatures [1].

However, operating an evaporator at elevated temperatures has the potential for severe scaling on heat exchanger surfaces because the solubility of some salts decreases at high temperatures (i.e., inverse solubility). Scale significantly reduces the thermal conductivity of heat transfer surfaces [2] and severely lowers performance. In thermal seawater desalination plants, the main technique currently employed to control alkaline scales – such as calcium carbonate ( $\text{CaCO}_3$ ) and magnesium hydroxide ( $\text{Mg}(\text{OH})_2$ ) – is the addition of antiscalants [2–5]. Their precipitates are soft and readily removed from heat exchanger surfaces by on-line acid washing [6] or sponge-ball cleaning [7,8]. To prevent non-alkaline scale such as calcium sulfate ( $\text{CaSO}_4$ ), the maximum top brine temperature is maintained below  $120^{\circ}\text{C}$ . From an economic viewpoint, operating evaporators at higher temperatures enhances performance substantially, provided scale is prevented [9].

In conventional MSF plants, the maximum brine temperature is limited by the  $\text{CaSO}_4$  barrier, which cannot be effectively controlled by antiscalants and cannot be easily removed by chemical or mechanical cleaning because of its hardness. Therefore, removing scale-forming constituents is the most effective means to prevent  $\text{CaSO}_4$  fouling, thus allowing evaporators to operate at higher temperatures. In general, three types of techniques are used to remove  $\text{Ca}^{2+}$  or  $\text{SO}_4^{2-}$ : lime-magnesium carbonate [10], nanofiltration (NF) [11–13], and ion exchange (IX) [14–22].  $\text{Ca}^{2+}$  and  $\text{HCO}_3^-$  can both be removed by reacting with  $\text{MgCO}_3$  and  $\text{Ca}(\text{OH})_2$ , respectively; the formed  $\text{CaCO}_3$  is filtered off. Although both alkaline and  $\text{CaSO}_4$  scale can be eliminated by the lime-magnesium carbonate process, the consumption of large amounts of chemicals has prevented its use as a pretreatment in desalination plants. NF membranes are used to specifically remove most divalent and some monovalent ions in seawater, depending on the type of membrane and operational conditions. As a seawater pretreatment to RO and MSF, NF was investigated in pilot plants [11,12]. When 90%

of divalent ions in seawater were removed, the maximum brine temperature could be increased as high as  $160^{\circ}\text{C}$  without using antiscalant [23]. Similar to RO, NF requires a series of pretreatment such as filtering the feed water before it goes to the membranes. However, thermal desalination is not so sensitive to suspended solids, therefore NF is excessive and costly when it is used as pretreatment for thermal desalination.

For decades, ion exchange (IX) has been proposed to reduce  $\text{Ca}^{2+}$  or  $\text{SO}_4^{2-}$  concentrations in seawater, using cationic or anionic resins, respectively. However, strong cationic resin show poor  $\text{Ca}^{2+}$  selectivity because of the high  $\text{Na}^+/\text{Ca}^{2+}$  ( $\sim 20$ ) and  $\text{Mg}^{2+}/\text{Ca}^{2+}$  ( $\sim 4.5$ ) ratio in seawater [14,15]. Weak anionic resins contain primary and secondary amino functional groups in a porous hydrophilic matrix and prefer monovalent ions at high total solution concentrations and polyvalent ions at low total solution concentrations [16]. This unique property allows this type of weak anionic resin to replace  $\text{SO}_4^{2-}$  from seawater feed with  $\text{Cl}^-$  from concentrated blow-down brine, thus saving the major cost of regenerant chemicals. The process of sulfate removal is called DESULF [17]. Using IX as pretreatment for MSF [17,18] and RO [19] has been satisfactorily demonstrated in desalination plants. The evaporators were operated at  $150^{\circ}\text{C}$  without forming  $\text{CaSO}_4$  scale [20,21]. During the IX exhaustion step, to achieve high selectivity of  $\text{SO}_4^{2-}$ , a large amount of acid was required to control seawater pH in the range of 3–4 [22]. IX and regeneration are usually run at slow speed to achieve satisfactory extent of sulfate removal, making IX expensive from a practical viewpoint.

This paper investigates the effectiveness of weak anionic resin (Relite MG1/P) on sulfate removal at various operational conditions. This resin has similar polyamine functional groups and polyacrylic matrix to those resins described in the literature and it can be regenerated with concentrated acidified (pH 4) blowdown brine. This study includes the effects of feed seawater pH, desalination concentration factors, and seawater flow rate on the degree of sulfate removal from seawater. To ensure the maximum brine temperature and production efficiency in thermal desalination, the degree of sulfate removal can be controlled by adjusting seawater flow rate. An economic evaluation of DESULF is presented at various maximum brine temperatures from  $140$  to  $180^{\circ}\text{C}$ .

## 2. Materials and methods

This study used the weak-base anion exchange resin Relite MG1/P (Mitsubishi, manufactured in



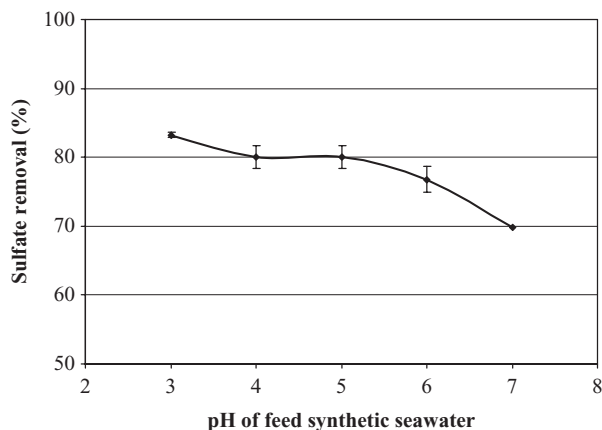


Fig. 1. Influence of pH on sulfate removal of synthetic seawater. Resin: Relite MG-1;  $F = 32$  L seawater/(L resin·h);  $V_{\text{ex}}/V_r = 16$  L seawater/L resin;  $V_{\text{reg}} = V_{\text{ex}}/n$ ;  $n = 2$ ; pH of regenerant = 4. Exhaustion or regeneration time = 30 min. The bar symbol represents  $\pm 1$  standard deviation.

This polyamine hydrolysis reaction occurs until the resin has attained pH equilibrium with water, decreasing the useful exchange capacity for sulfate removal [18].

To reverse the hydrolysis reaction (Reaction 4) during regeneration, acid was added to the regenerant to convert the hydrolyzed group into the chloride form. Fig. 1 shows the effect of pH on the extent of sulfate removal in synthetic seawater with a concentration factor of 2. Each point represents the average of several consecutive cycles of the steady-state sulfate removal. Lowering the pH of feed water favored sulfate removal. It is apparent that the extent of sulfate removal remained constant within a pH range of 4–5, and then decreased significantly. Furthermore, the resin performance continuously decreased even after seven cycles when synthetic seawater at pH 7 was used. These results agree with the literature. In synthetic pH 7 seawater, bicarbonate ions consume protons to obtain pH 5 buffer, favoring polyamine hydrolysis (Reaction 4), consequently decreasing the resin exchange capacity. In industry, pH 5 effluent would be sent to a degasifier to remove bicarbonate as  $\text{CO}_2$  gas. Therefore, adjusting seawater pH to 4–5 ensures the weak-base anion exchange resin has high exchange capacity for removing sulfate.

### 3.2. Concentration factor

Fig. 2 demonstrates the effects of concentration factor  $n$  (i.e., the concentration ratio of blowdown brine to feed water) on sulfate removal in synthetic seawater. Increasing  $n$  improves sulfate removal because it delivers dual benefits to the IX process. Large  $n$  mean high

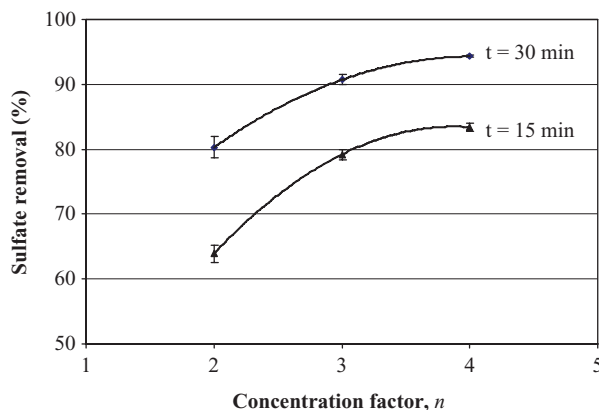


Fig. 2. Effect of concentration factor on sulfate removal of synthetic seawater. Resin: Relite MG-1;  $V_{\text{reg}} = V_{\text{ex}}/n$ ; pH of feed synthetic seawater = 4–5. pH of regenerant = 4. Exhaustion or regeneration time = 15 or 30 min. The bar symbol represents  $\pm 1$  standard deviation.

$\text{Cl}^-$  concentrations in the regenerant, therefore reducing the resin preference for  $\text{SO}_4^{2-}$  and helping substitute  $\text{SO}_4^{2-}$  with  $\text{Cl}^-$  during the regeneration process. Moreover, using equal times for exhaustion and regeneration, the volume and flow rate of regenerant are  $1/n$  those of the exhaustion process. Because regeneration efficiency strongly depends on the liquid-solid contact time, a high concentration factor means a slow regenerant flow rate and a high regeneration efficiency. By the same reasoning, reducing  $n$  resulted in worse sulfate removal at higher flow rate (Fig. 2). From a practical viewpoint, higher  $n$  means less seawater must be treated and less pretreatment cost per volume of produced water. On the other hand, the use of higher  $n$  requires more energy in the distillation process and increases the potential for scaling on the distillation heat exchangers. Therefore, selecting an appropriate  $n$  requires optimization of the whole desalination technology.

During regeneration, there is an issue of  $\text{CaSO}_4$  precipitation formation because of high  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  concentration in the regeneration effluent. Ion exchange and regeneration processes were performed at room temperature (i.e., 20–25°C);  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (gypsum) is the major precipitant. It has been reported that solubility of  $\text{CaSO}_4$  in water increases in the presence of  $\text{NaCl}$ . For example, at 25°C, experimental data showed that gypsum solubility in 1-N and 2.6-N  $\text{NaCl}$  solutions is 0.045 and 0.0568 mol/L, respectively [27]. In the regeneration effluent, Table 2 presents the  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  concentrations and the  $\text{CaSO}_4$  solubility product ( $K_{\text{SP}}$ ), which was obtained by correlating data published by Bock [27]. Because only  $\text{SO}_4^{2-}$  was removed during IX, the  $\text{Ca}^{2+}$  concentration in the regeneration effluent is 2–4 times of that in the exhaustion effluent.

Table 2  
CaSO<sub>4</sub> K<sub>SP</sub> in the effluent of regeneration with various concentration factor at 25°C

	Seawater	Regeneration effluent		
		Concentration factor		
		2	3	4
NaCl (mol/L)	0.054	1.2	1.8	2.4
Ca <sup>2+</sup> (mol/L)	0.0104	0.0208	0.0312	0.0416
SO <sub>4</sub> <sup>2-</sup> (mol/L)	0.0281	0.04496	0.06744	0.08992
CaSO <sub>4</sub> K <sub>SP</sub> from regeneration effluent	2.9E-4	9.3E-4	2.1E-3	3.7E-3
CaSO <sub>4</sub> K <sub>SP</sub> in NaCl solution	1.3E-3	2.3E-3	2.9E-3	3.2E-3

The last 25% of the exhausted regenerant was used as the first fraction in the subsequent regeneration. At equilibrium, the SO<sub>4</sub><sup>2-</sup> concentration in the regeneration effluent was estimated as the total SO<sub>4</sub><sup>2-</sup> in treated seawater divided by the total volume of regenerant. With a concentration factor of 2 and 3, Table 2 shows that the CaSO<sub>4</sub> K<sub>SP</sub> in the regeneration effluent is smaller than K<sub>SP</sub> in the corresponding NaCl solution [27], thus indicating that no CaSO<sub>4</sub>·2H<sub>2</sub>O (gypsum) is formed during regeneration. However, a high concentration factor means that a small volume of regenerant has high Ca<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup> in the regeneration effluent, leading to a higher CaSO<sub>4</sub> K<sub>SP</sub> in the regeneration effluent than that in the corresponding NaCl solution [27]. Therefore, with concentration factor of 4, CaSO<sub>4</sub>·2H<sub>2</sub>O precipitants might form during regeneration. Because of the potential for precipitant formation, a concentration factor of 3–3.5 is recommended in the literature [21].

### 3.3. Exhaustion flow rate

Relite MG1/P is an ideal choice because it has a high selectivity towards SO<sub>4</sub><sup>2-</sup> at seawater concentrations and preference for Cl<sup>-</sup> at higher solution concentrations. Other important characteristics are its high exchange capacity and fast exchange rate, similar to those of Kastel A 102. We assumed that Relite MG1/P and Kastel A 102 have the same bed volume of 40 L water/(L resin·h) [18]. As discussed above, regeneration and exhaustion are closely coupled when using equal processing times. A high exhaustion flow rate means a high regenerant flow rate and relatively low regeneration efficiency, consequently worsening the subsequent cycle. To obtain high regeneration efficiency, the last 25–30% of the exhausted regenerant was used as the first fraction in the subsequent regeneration. Furthermore, fixed-bed IX was operated

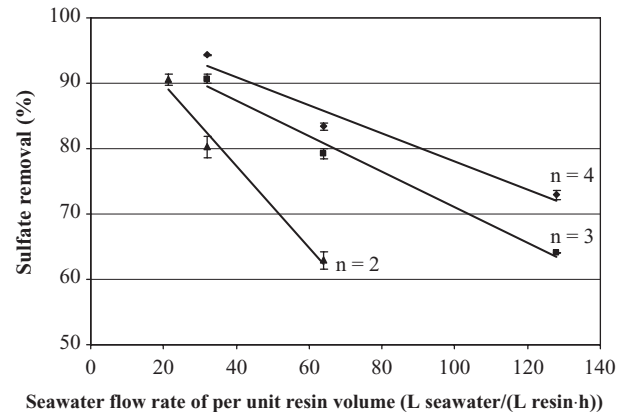


Fig. 3. Sulfate removal versus flow rate at various concentration factors. Resin: Relite MG-1/P;  $V_{\text{ex}}/V_r = 16$  L seawater/L resin;  $V_{\text{reg}} = V_{\text{ex}}/n$ . pH of regenerant = 4. The bar symbol represents  $\pm 1$  standard deviation.

countercurrently, i.e., downflow regeneration with upflow exhaustion was employed to utilize the regenerant efficiently.

In practice, increasing the flow rate is useful when a high degree of sulfate removal is not required. High flow rates reduce the equipment size and the quantity of resin, thus lowering capital cost. Fig. 3 demonstrates the extent of sulfate removal with various exhaustion times and concentration factors. The IX times of the desalination process with  $n$  values of 3 and 4 were 7.5, 15, and 30 min. In this study, with a 30-min exhaustion process and a corresponding specific volume of 16 L water/L resin, the bed volume varies from 32 to 128 L seawater/(L resin·h). To obtain a high extent of sulfate removal with a concentration factor of 2, IX was run at a lower flow rate, i.e., longer IX and regeneration times of 15, 30, and 45 min. Fig. 3 shows the inverse linear correlation of sulfate removal and flow rate with  $n$  varying from 2 to 4. With smaller values of  $n$ , the lines have larger absolute values of the slope, meaning that the extent of sulfate removal decreased more drastically at higher flow rates with lower concentration factors  $n$ . The difference in sulfate removal resulting from the change of concentration factor became smaller at lower flow rates. Thus, high flow rates favor high concentration factors; low concentration factors require low flow rates to attain the required sulfate removal. For a given  $n$ , the linear correlation helps to determine the flow rate or IX times required to achieve the desired degree of sulfate removal. Moreover, the close association of concentration factor and flow rate must be considered when optimizing IX and distillation processes from economical and practical viewpoints.

Table 3  
Solubility limits of calcium sulfate hemihydrate  $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$  (mg  $\text{SO}_4/\text{L}$ )

Maximum brine $T$ ( $^{\circ}\text{C}$ )	Maximum feed water concentration of $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$ (mg $\text{SO}_4/\text{L}$ )						Minimum sulfate removal* (%)					
	Concentration factor						Concentration factor					
	1	2	2.5	3	3.5	4	1	2	2.5	3	3.5	4
120	6,197	2,979	2,160	1,494	1,058	743	0	0	20	45	61	73
130	4,166	1,999	1,450	1,004	711	500	0	26	46	63	74	82
140	2,801	1,368	992	674	478	336	0	49	63	75	82	88
150	1,883	938	680	453	322	226	30	65	75	83	88	92
160	1,266	643	466	304	216	152	53	76	83	89	92	94
170	851	440	319	204	145	102	68	84	88	92	95	96
180	572	302	219	137	98	69	78	89	92	95	96	97

\*Based on sulfate content in seawater: 2,700 ppm.

#### 4. Economic evaluation

Because the solubility of  $\text{CaSO}_4$  decreases as temperature increases (i.e., inverse solubility), a high degree of sulfate removal is required for seawater fed to evaporators operating at high temperatures. A high concentration factor, leading to high  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$ , can also precipitate  $\text{CaSO}_4$ . Therefore, the extent of sulfate removal required to prevent  $\text{CaSO}_4$  formation on heat exchangers highly depends on both operating temperatures and concentration factors. For a given maximum brine temperature and concentration factor, Table 3 summarizes the maximum allowable concentration of  $\text{SO}_4^{2-}$  fed to evaporators or the minimum sulfate removal required to prevent  $\text{CaSO}_4$  scaling. The data were obtained by correlating and extrapolating data published by Zannoni et al. [19]. Sulfate removal is not necessary with concentration factors below 2 and temperatures below  $120^{\circ}\text{C}$ , which are parameters currently employed in MSF desalination plants. Above  $120^{\circ}\text{C}$  or with  $n$  values above 2, sulfate removal is required to prevent  $\text{CaSO}_4$  fouling.

For a fixed quantity of produced water, the amount of treated seawater depends only on the adopted value of  $n$ . However, the required amount of resin, as well as the size of the DESULF equipment, depends on both  $n$  and the evaporator operating temperature. Lower  $n$  or lower operating temperatures require less sulfate removal, implying less resin and smaller equipment. Higher  $n$  needs less feed seawater into DESULF unit per volume of produced water and produces less volume of regenerant; thus, higher flow rates can be used in the DESULF process to reduce the amount of resin and the size of pretreatment equipment.

On the basis of the design parameters discussed above, the whole DESULF flow sheet is illustrated in Fig. 4. To prevent calcium sulfate formation in heat exchangers operating at  $180^{\circ}\text{C}$ , Table 3 shows that 90%,

95%, and 97% of seawater sulfate should be removed with concentration factors of 2, 3, and 4, respectively. As shown in Fig. 3, decreasing the liquid flow rates or increasing the concentration factor enhance sulfate removal, thus allowing the evaporators to operate at  $180^{\circ}\text{C}$ .

To be consistent with the novel high-temperature MVC desalination system [1], the concentration factor remained constant (i.e., 2) with temperatures ranging from  $140$  to  $180^{\circ}\text{C}$ . Table 4 summarizes the cost of a desalination plant with a production capacity of 1 million gallons per day (MGD). The cost of DESULF pretreatment includes resin (Relite MG1/P,  $\$180/\text{ft}^3$ ) and necessary equipment, as shown within the dotted line in Fig. 4. The required amount of resin and the size of equipment were estimated from the sulfate removal data in Fig. 3. Similarly, the pretreatment cost with concentration factors of 3 and 4 can be evaluated with the same method. The equipment cost was evaluated by ASPEN ICARUS. Despite the high cycle frequency, the resin consumption was about 15% per year because of small osmotic shocks and low volume variations during the exhaustion and regeneration steps [20]. The capital cost varies from  $\$0.068$  to  $\$0.178$ /thous gallon, whereas the operating cost remains the same as  $\$0.178$ /thous gallon with temperature changes from  $140$  to  $180^{\circ}\text{C}$ . The total pretreatment cost, summation of capital cost and operating cost, ranges from  $\$0.246$  to  $\$0.356$ /thous gallon produced water. Because resin replacement accounts for 50% of the operating cost, a less costly weak base anion exchange resin (A454G, Mitsubishi, manufactured in China,  $\$120/\text{ft}^3$ ) could substitute for Relite MG1/P. The estimated production cost of seawater desalination with the novel high-temperature MVC [1] is  $\$1.44$  and  $\$1.86$ /thous gallon in the Middle East and United States, respectively; therefore, the DESULF pretreatment cost is about 20% of the total production cost. The novel high-temperature MVC economics are attractive

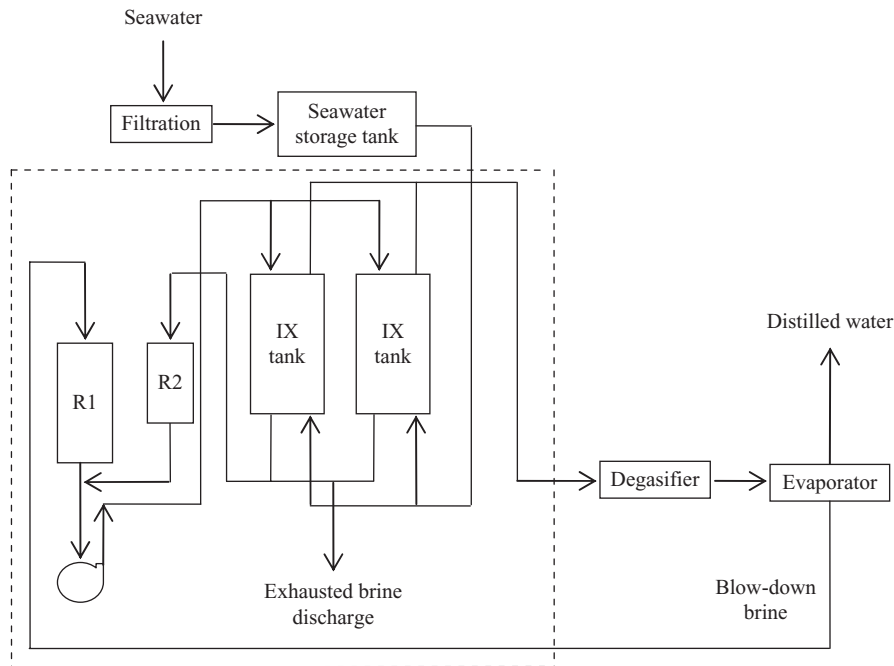


Fig. 4. Flow sheet of DESULF unit R1, R2 = Regenerant tanks.

compared with traditional seawater desalination technologies of MSF (\$2.92–5.95/thous gallon) and RO (\$2.42–8.98/thous gallon) [28] depending on the capacity.

**5. Conclusions**

Laboratory studies have demonstrated that sulfate can be removed from seawater acidified at pH 4–5 by

using weak-base anion exchange resin. The resin can be regenerated by concentrated acidified (pH 4) blowdown brine, consequently saving the cost of regenerants. The concentration factor significantly affects sulfate removal at higher liquid flow rates. With a fixed concentration factor, each maximum brine temperature requires different extents of sulfate removal, which can be achieved by adjusting the feed water flow rate. The flow rate determines the required amount of resin and the equipment

Table 4  
Cost evaluation of desulfation units using Relite MG 1/P with concentration factor of 2

		Maximum brine temperature (°C)				
		140	150	160	170	180
Pretreatment cost						
Capital cost	Resin cost (\$)	44,100	63,000	94,500	133,200	189,000
	Fixed capital investment <sup>①</sup> (\$)	270,600	315,300	404,800	495,600	640,660
	Summation of capital cost (\$)	314,700	378,300	499,300	628,800	829,660
	Amortization life (year)	30	30	30	30	30
	Interest (%)	5	5	5	5	5
	Capital cost (\$/thous gal produced water)	0.068	0.081	0.107	0.135	0.178
Operating cost	Resin replacement	0.06	0.06	0.06	0.06	0.06
	Acid treatment (\$/thous gal produced water)	0.05	0.05	0.05	0.05	0.05
	Electricity <sup>②</sup> (\$/thous gal produced water)	0.038	0.038	0.038	0.038	0.038
	Labor (\$/thous gal produced water)	0.03	0.03	0.03	0.03	0.03
	Total operating cost (\$/kgal produced water)	0.178	0.178	0.178	0.178	0.178
Total pretreatment cost (\$/ thous gal produced water)		0.246	0.259	0.295	0.313	0.356

<sup>①</sup> 6 times equipment cost.

<sup>②</sup> Energy consumption: 0.1 kWh/m<sup>3</sup>.

Electricity cost: \$0.10/kWh.

size. For a concentration factor of 2, economic evaluation (including resin and equipment cost) was performed at various maximum brine temperatures. For a desalination plant with a production capacity of 1 MGD, the cost of DESULF pretreatment varies from \$0.246 to \$0.356/thous gallon produced water as the maximum temperature changes from 140 to 180°C, respectively. The DESULF process allows for appreciable savings in the distillation system by raising the operating temperature, which allows for high heat transfer coefficients and reduced compressor size.

### Symbols

$F$	specific flow rate, L/h
$K_{SP}$	solubility product
meq	milliequivalent
$n$	concentration factor between discharged brine and feed water of desalination plant, $(\text{kg}/\text{m}^3)_{\text{out}}/(\text{kg}/\text{m}^3)_{\text{in}}$
$T$	temperature, °C
$V_{\text{ex}}$	treated water volume, L
$V_{\text{reg}}$	regenerant volume, L
$V_{\text{r}}$	resin bed volume, L
$V_{\text{ex}}/V_{\text{r}}$	specific volume of treated water, L water/L resin

### Subscripts

ex	exhaustion
reg	regeneration

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