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The effect of temperature, ion valency, and solvent reuse on the solvent extraction desalination

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Abstract. This study investigates the performance of solvent extraction desalination (SED) using three amine solvents DPA, DIPA, and DBA under various temperature and ion valency conditions. The results revealed that reaction and separation temperatures significantly influenced water recovery and salt removal efficiencies. DIPA demonstrated the highest water recovery, while DPA exhibited superior salt removal efficiency. Mg^{2+} was effectively removed through a precipitation mechanism, distinct from the polarity-driven removal of sodium chloride. Additionally, reuse experiments over nine cycles showed stable performance in water recovery and salt removal, emphasizing the economic and operational feasibility of SED. These findings highlight the adaptability and potential of SED as a membrane-free desalination technology for high-brine scenarios, offering a sustainable alternative for seawater and brine treatment.

Keywords: amine solvents (DPA, DIPA, DBA); magnesium ion (Mg^{2+}) removal; solvent extraction desalination; salt removal efficiency; solvent reuse; water recovery efficiency

1. Introduction

Solvent extraction, also known as liquid-liquid extraction, is a method of separating compounds based on their relative solubility in different immiscible liquids, usually water (polar) and an organic solvent (non-polar). This method has been applied across various fields, including the food industry, chemistry, and metal extraction (Filiz *et al.* 2006, Patel and Karamalidis, 2021, Wang *et al.* 2021). It has also been utilized in desalination, emerging as an alternative for traditional desalination (Barbosa *et al.* 2021, 2022, Choi *et al.* 2021, Boo *et al.* 2019, Alotaibi *et al.* 2017, Sanap *et al.* 2015, Bajpayee *et al.* 2011, Luo *et al.* 2011). Although solvent extraction desalination (SED) was first proposed by Davidson *et al.* (1960) with secondary and tertiary amines, the study was limited to desalination below 5,000 mg/L of salinity. To overcome the limitation of the study of Davidson *et al.* (1960), Bajpayee *et al.* (2011) showed that SED was possible by combining carboxylic acids and fatty acids with saltwater using decanoic and hexanoic acids. However, Alotaibi *et al.* (2017) suggested by modeling that use of decanoic acid and octanoic acid results in low yield and high water solubility and consequently not suitable for SED. In addition, Sanap *et al.* (2015) showed that this process consumes significantly more resources per unit of salt-free water produced than alternatives and well-established desalination technologies which would increase the treatment cost.

Choi *et al.* (2019) evaluated the performance of SED using three non-aqueous solvents (NAS) with different molecular structures and demonstrated their effectiveness through molecular dynamics (MD) simulations. This study provided valuable insights into the mechanisms governing the interactions between water, salt ions, and NAS. In a subsequent study, among seven different primary and secondary amines tested, Dipropylamine (DPA) exhibited the highest desalination performance, demonstrating its competitiveness with conventional thermal processes (Choi *et al.* 2021). Similarly, Boo *et al.* (2019) investigated the performance of SED using three solvents and further demonstrated that high-salinity brine (5.0 M NaCl) desalination was feasible with Diisopropylamine (DIPA).

Successive studies on solvent properties, primarily using simulations, significantly contributed to understanding the fundamental principles of SED. Kim *et al.* (2020) identified surface polarity as a key factor influencing SED performance in three organic solvents. Barbosa *et al.* (2021) further demonstrated that the NH group in DIPA enhances bonding with water molecules, utilizing DIPA and imidazole-based solvents. Additionally, a comparative analysis of the water solubility of four amine solvents, conducted via interfacial simulations, confirmed that DPA exhibited the highest performance among them.

Although the desalination performance of DIPA and DPA has been investigated in many papers, there are few studies using seawater. Although NaCl is the primary component of seawater, magnesium ions (Mg^{2+}) can impact desalination performance by potentially hindering the adsorption and ion exchange of organic acids (Balistrieri and Murray 1987, Inglezakis *et al.* 2005, Zuo *et al.* 2013).

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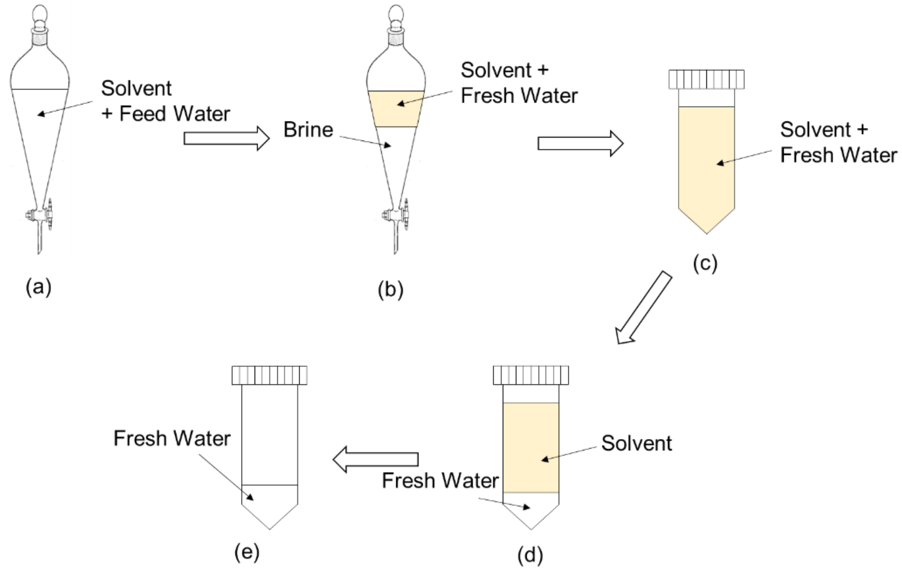
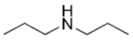
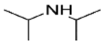
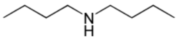


Fig. 1 Flow diagram of a process for extractive solvent desalination

Table 1 Chemical properties of amine compounds as desalination solvent

	DPA	DIPA	DBA
Chemical structure			
Chemical formula	$C_6H_{15}N$	$C_6H_{15}N$	$C_8H_{19}N$
Molecular weight (g/mol)	101.19	101.19	129.247
Density (g/mL)	0.738	0.717	0.767
Boiling point (°C)	109	83-85	137-177
Solubility (g/L)	35.1	10	4.7
log K_{ow}	1.7	1.4	2.8
Purity	> 99 %	>99.5 %	> 99.5%

Mg^{2+} is the fourth most abundant ion in seawater and should be considered when applying to seawater desalination. This study conducted SED using DPA, DIPA, and DBA (Dibutylamine) on artificial seawater and $MgCl_2$ solution. The objective was to compare the treatment efficiency under different conditions and to demonstrate the changes in efficiency when the solvent was reused.

2. Materials and methods

2.1 Solvent extraction desalination process

Two synthetic seawater solutions, each with a total dissolved solids concentration of 3.5%, were prepared by dissolving sea salt (Sigma Aldrich, USA) and magnesium chloride ($MgCl_2$, Samchun Chemicals, Korea) in deionized water. The SED process consists of mixing and separation steps. SED procedure consists of mixing and separation process. The mixing temperatures of the solvent and aqueous solutions were set to 5°C, 15°C, and 25°C, and the separation temperature were set to 45°C, 65°C, and 85°C. As shown in Fig. 1, pre-heated solvent (200 ml) and feed water (200 ml) solution at 5°C, 15°C, and 25°C were filled into a 500 mL separatory funnel, and then mixed vigorously by handshaking for 1 min (Fig. 1(a)). The mixture was separated into brine and solvent mixture (solvent and freshwater) based on density and polarity after holding the mixture still for 5 minutes (Fig. 1(b)). The brine was removed from the bottom of a separatory funnel and solvent mixture was taken into a test tube. To promote separation, the solvent mixture was heated and then kept in 45°C, 65°C, and 85°C for 1 hour in an oven. The solvent mixture was heated and then kept in 45°C, 65°C, and 85°C for 1 hour in an oven. The solvent mixture was separated into solvent (top layer) and fresh water (bottom layer) on an oven (Fig. 1(d)). Finally, fresh water was carefully sampled from the bottom layer, after which fresh water volume and ion concentrations of the aqueous solution were measured. The concentration of total ions was measured using Ion Chromatography (ICS-900, Dionex, USA). The concentration of Mg^{2+} ions was analyzed using an Atomic Absorption Spectroscopy (AAS, Thermo Scientific, USA). For the solvent reuse experiment, the procedures were repeated using the remaining solvent after fresh water was taken.

2.2 Solvent properties

Table 1 shows chemical properties of DPA, DIPA, and DBA (Tokyo Chemical Industry, Japan).

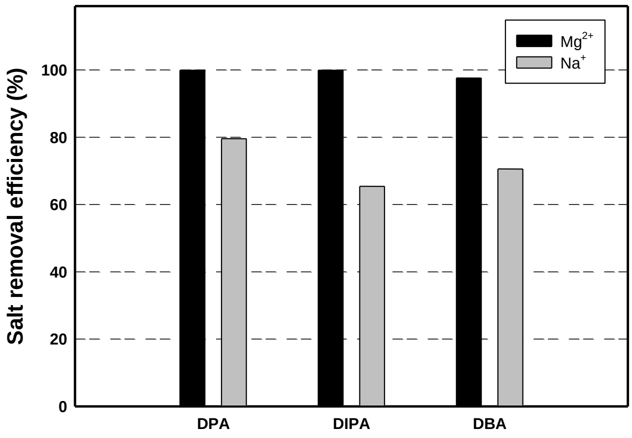


Fig. 2 Comparison of Na^+ and Mg^{2+} removal efficiency for DPA, DIPA and DBA

2.3 Analytical methods

The SED was evaluated by means of the water recovery rate and the salt removal efficiency. The water recovery rate is defined as the ratio of volume of recovered fresh water and the total volume of feed water as shown in Eq. (1). The salt and Mg^{2+} removal efficiencies are calculated based on the difference in ion concentration between the feed water and the fresh water, as shown in Eq. (2).

$$\begin{aligned}&\text{Water recovery efficiency (\%)}\\&= \frac{Volume\ of\ Fresh\ Water(ml)}{Volume\ of\ Feed\ Water(ml)} \times 100\end{aligned} \tag{1}$$

$$\begin{aligned}&\text{Salt removal efficiency(\%)}\\&= [1 - \frac{Salt\ concentration\ of\ Fresh\ Water}{Salt\ concentration\ of\ Feed\ Water}] \times 100\end{aligned} \tag{2}$$

3. Results and discussion

3.1 Mg^{2+} Removal using SED

Mg^{2+} removal using the SED was carried out at the three different reaction temperatures and three different separation temperatures. After treating 3.5% $MgCl_2$ using SED, the Mg^{2+} concentration ranged from 1.0 to 9.0 mg/L with DPA, 1.0 to 8.5 mg/L with DIPA, and 215 to 356.5 mg/L with DBA. The removal efficiency for DPA and DIPA was 99.9%, indicating that most of the Mg^{2+} was removed under all conditions, while DBA showed a relatively lower removal efficiency of 96.0–97.6% compared to the other two solvents. Among the tested conditions, a reaction temperature of 25°C and a separation temperature of 85°C achieved the highest performance, with Mg^{2+} removal efficiencies of 99.9% for DPA and DIPA and 97.6% for DBA, as shown in Fig. 2. These values significantly exceeded the NaCl removal rates reported in previous studies (Bajpayee *et al.* 2011, Boo *et al.* 2019, Choi *et al.* 2021). Mg^{2+} was easily removed using SED, indicating a distinct removal mechanism different from that of NaCl in the SED process. Other studies using SED have also reported that Mg^{2+} is effectively removed (Gouda *et al.* 1993). The NaCl removal of the SED is related to the polarity of the cluster surface, whereas Mg^{2+} is related to the precipitation and dissolution. As shown in Eq. (3), amines can be combined by extracting H^+ from water to the organic phase, leaving OH^- in the aqueous phase (Wang *et al.* 2015, Said *et al.* 2020). In the aqueous phase, Mg^{2+} mostly exists in an ionic state and combines with OH^- generated in Eq. (3) to form $Mg(OH)_2$ (Eq. (4)). Similar precipitation and dissolution reaction of Mg^{2+} and amine in the aqueous phase was used for lactic acid extraction, and there was no additional reaction between Mg^{2+} and amine (Van Krieken 2011). Removing Mg^{2+} through precipitation leads to greater amine loss as Mg^{2+} concentration increases. Additionally, since the precipitation reaction occurs before NaCl absorption, it may reduce the efficiency of SED.

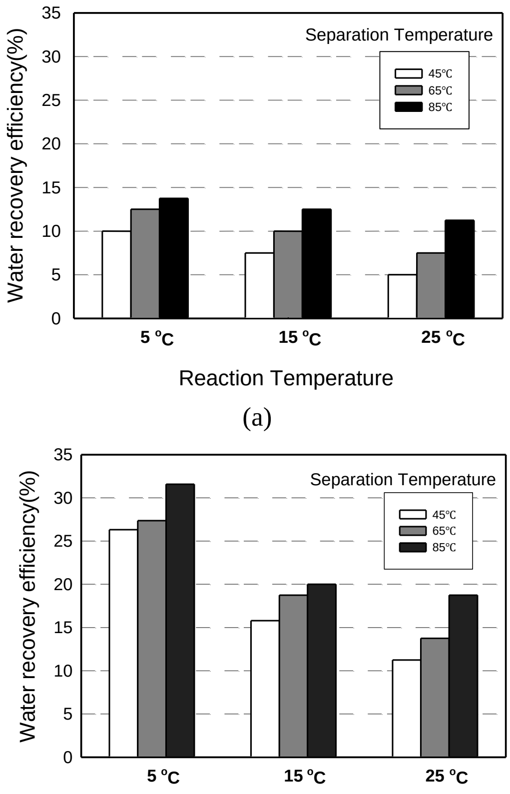
$$\mathrm{H_2O + C_6H_{15}N \rightleftharpoons C_6H_{16}N^+ + OH^-} \tag{3}$$

$$\mathrm{OH^- + Mg^{2+} \rightleftharpoons Mg(OH)_2} \tag{4}$$

3.2 Desalination of synthetic seawater

The SED experiments with 3.5% synthetic saltwater were carried out with DPA, and DIPA at various temperatures. Water recovery efficiency for DPA and DIPA are shown in Fig. 3. These results were consistent with temperature-dependent solubility properties of secondary amines (Davidson *et al.* 1960, Stephenson, 1993). The water recovery efficiency of DIPA was higher than that of DPA, consistent with previous studies using NaCl solution (Boo *et al.* 2019, Choi *et al.* 2021). Additionally, the efficiency exhibited different trends depending on the reaction and separation temperatures. When using DIPA, the water recovery was 31.6% at a reaction temperature of 5°C and a separation temperature of 85°C. However, as the reaction temperature increased to 25°C, the recovery significantly decreased to 18.8%. In contrast, DPA showed minimal variation under the same conditions, with water recovery changing only slightly from 13.8% to 12.5%. At each reaction temperature, the highest water recovery efficiency was not much changed for DPA (i.e., 1.2 times), but DIPA showed a rather large difference (1.7 times).

Water recovery efficiencies tended to decrease as the octanol-water partition coefficient (K_{ow}) and carbon chain length increased (Choi *et al.* 2021). The water recovery efficiencies are affected by the carbon chain length even if they have the same chemical formula (Choi *et al.* 2021). The length of the carbon chain makes difference in basicity. The basicity of a molecule is a measure of its ability to accept protons (H^+) to form a stable product, and the property varies with the length of the carbon chain (Fitzsimons *et al.* 2012, Pontes 2018). The difference of DPA and DIPA are thought to be affected by basicity, which is the ability to dissolve water by forming hydrogen bonds. This means that DIPA with high basicity due to a short carbon chain length accepts more water than DPA. This explains why DIPA shows better water recovery efficiency.



(a)

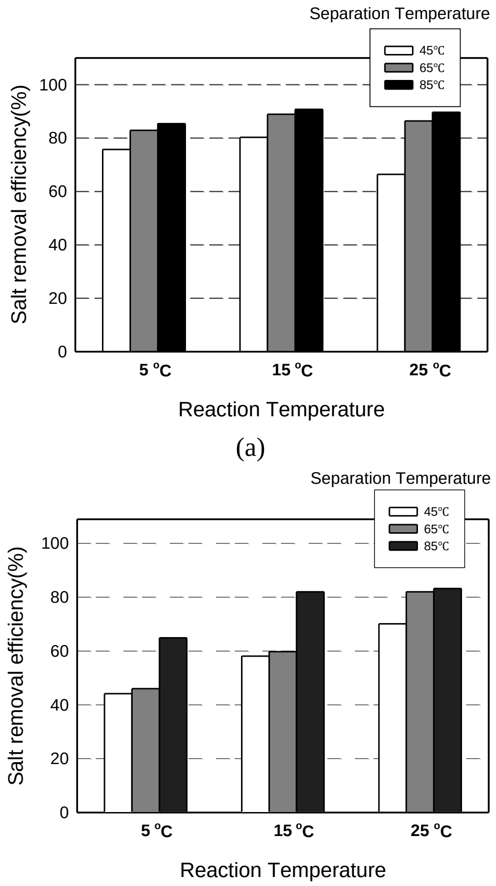
(b)

Fig. 3 Water recovery efficiencies for DPA (a) and DIPA (b)

Fig. 4 shows the salt removal efficiency of the DPA and DIPA solvents according to reaction and separation temperatures. The salt removal efficiency increased as the reaction and separation temperature increased except for one case as shown at the Fig. 4. DPA had the highest salt removal efficiency of 90% at 15°C reaction temperature and 85°C separation temperature. DIPA had the highest salt removal efficiency of 83. % at 25°C reaction temperature and 85°C separation temperature. Each showed about 3% lower performance than the experiment using the previous NaCl solution (Boo *et al.* 2019, Choi *et al.* 2021). The SED using seawater did not show significant difference with the results of NaCl aqueous solution. This might be because Mg^{2+} in seawater is removed by precipitation mechanism and thus does not affect the SED salt removal efficiency significantly. In addition, the other ions concentration shall be considerably lower than that of Mg^{2+}, and thus they could not affects the SED efficiency significantly either. Therefore, NaCl removal in the SED is critical for the salt removal efficiency.

3.3 Solvent reuse

Reuse of solvent is a critical factor in the aspect of economic feasibility. Nine SED was repeated using reused DPA and artificial saltwater. DPA was selected as the solvent as it exhibited higher salt removal efficiency. The reaction and separation temperatures were chosen as the highest salt removal rate from the previous experiments (i.e., reaction temperature 15°C, separation temperature 85°C). Solvent reuse does not seem to reduce desalination efficiencies during nine repeated experiments as shown at Fig. 5. The water recovery efficiency was about 12 ± 0.5%, and the salt removal efficiency was maintained at about 89 ± 1.85%. If the solvent can be reused, about 0.93 tons of solvent is required when producing 1 ton of fresh water, but if not reusable, about 8.4 tons is required. Therefore,



(a)

(b)

Fig. 4 Salt removal efficiencies for DPA (a) and DIPA (b)

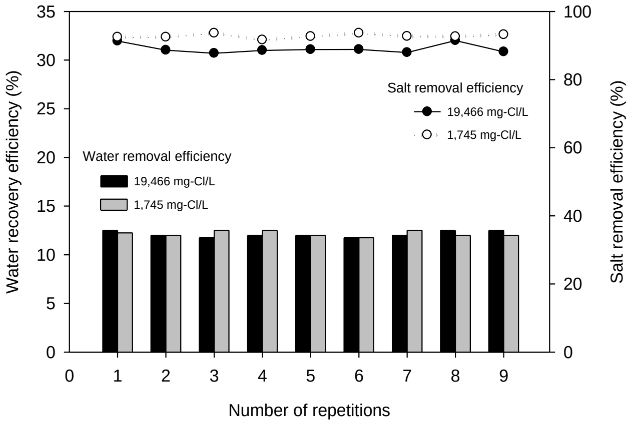


Fig. 5 Water recovery and salt removal efficiencies over solvent reuse

solvent reusing is critical for the economic feasibility of the SED process.

Boo *et al*. (2019) installed continuous SED with solvent reuse but water recovery efficiency decreased with DIPA (2019). The difference of the water recovery rate could be explained by the process difference by which fresh water is produced. Desalination using DPA showed stable efficiency at a constant mixing and separation temperature. This is because the solubility of salt and water, inherent to DPA's unique properties, is the primary factor determining freshwater production. Therefore, if the temperature conditions for desalination remain the same, the solvent can be continuously used (Davidson *et al*. 1960, Bajpayee *et al*. 2011, Luo *et al*. 2011). Furthermore, experiments on the reaction between NaCl and the organic solvent revealed that NaCl ions bind to the surface of the solvent clusters at lower temperatures and then separate at higher temperatures. This also provides evidence supporting the feasibility of repeated desalination using DPA (Kim *et al*. 2020).

4. Conclusions

This study explored the potential of SED using various amines, particularly DPA, DIPA, and DBA, under different temperature conditions and ion valency settings. The findings confirmed that the performance of SED is significantly influenced by the reaction and separation temperatures. DIPA demonstrated superior water recovery efficiency, while DPA achieved higher salt removal rates. The study showed DPA can be reused at least 9 times and yet maintain the salt removal efficiency. The ability of the solvents to maintain desalination performance even with repeated reuse underscores the economic and operational viability of the process. Moreover, the removal efficiency of magnesium ions (Mg^{2+}), often regarded as a challenge in seawater desalination, was consistently high, demonstrating an alternative removal mechanism through precipitation. This highlights the adaptability of SED to diverse ion compositions, offering a robust solution for treating complex saline water.

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