Fates of water and salts in non-aqueous solvents for directional solvent extraction desalination: Effects of chemical structures of the solvents

Ohkyung Choi^{1a}, Minsup Kim^{2a}, Art E. Cho², Young Chul Choi³, Gyu Dong Kim⁴, Dooil Kim⁵ and Jae Woo Lee^{*1}

¹Department of Environmental Engineering, Korea University, Sejong 30019, Republic of Korea
²Department of Bioinformatics, Korea University, Sejong 30019, Republic of Korea
³Water Research Center, Southern Research, GA 30120, USA
⁴Energy, Environment & Engineering, RTI International, NC 27709, USA
⁵Department of Civil and Environmental Engineering, Dankook University, Yongin 16890, Republic of Korea

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Abstract. Non-aqueous solvents (NASs) are generally known to be barely miscible, and reactive with polar compounds, such as water. However, water can interact with some NASs, which can be used as a new means for water recovery from saline water. This study explored the fate of water and salt in NAS, when saline water is mixed with NAS. Three amine solvents were selected as NAS. They had the same molecular formula, but were differentiated by their molecular structures, as follows: 1) NAS 'A' having the hydrophilic group (NH₂) at the end of the straight carbon chain, 2) NAS 'B' with symmetrical structure and having the hydrophilic group (NH) at the middle of the straight carbon chain, 3) NAS 'C' having the hydrophilic group (NH₂) at the end of the straight carbon chain but possessing a hydrophobic ethyl branch in the middle of the structure. In batch experiments, 0.5 M NaCl water was blended with NASs, and then water and salt content in the NAS were individually measured. Water absorption efficiencies by NAS 'B' and 'C' were 3.8 and 10.7%, respectively. However, salt rejection efficiency was 98.9% and 58.2%, respectively. NAS 'A' exhibited a higher water absorption efficiency of 35.6%, despite a worse salt rejection efficiency of 24.7%. Molecular dynamic (MD) simulation showed the different interactions of water and salts with each NAS. NAS 'A' formed lattice structured clusters, with the hydrophilic group located outside, and captured a large numbers of water molecules, together with salt ions, inside the cluster pockets. NAS 'B' formed a planar-shaped cluster, where only some water molecules, but no salt ions, migrated to the NAS cluster. NAS 'C', with an ethyl group branch, formed a cluster shaped similarly to that of 'B'; however, the boundary surface of the cluster looked higher than that of 'C', due to the branch structure in solvent. The MD simulation was helpful for understanding the experimental results for water absorption and salt rejection, by demonstrating the various interactions between water molecules and the salts, with the different NAS types.

Keywords: non-aqueous solvent (NAS); desalination; directional solvent extraction (DSE); salt rejection; molecular dynamics

1. Introduction

It is frequently noticed that climate change causes depletion of available freshwater resources and water scarcity in many regions (Amell 1999). It has been reported that about 40% of the global population lives in waterstressed areas (Marchal *et al.* 2011). Without securing alternative water resources, it has been assessed that 90% of the total available freshwater in the world will be used up by 2025 (Hoffmann 2009, Harrison 1997). Thus, global interest in securing water resources increases year by year. To promote water security, desalination technology has been developed over a long period, with great advances in recent decades. Costs have decreased accordingly, by approximately nine times since the 1950s, and the total capacity of seawater desalination plants is expected

E-mail: jaewoo@korea.ac.kr

to increase to more than 28 million m^3/day by 2025 (Zhou and Tol 2005).

Desalination technologies developed to date include evaporative processes, membrane processes, electrodialysis (ED), and capacitive deionization (CDI). Of these, multistage flash distillation (MSF), and reverse osmosis (RO), have been widely applied globally (Shannon et al. 2008, Service 2006). MSF is a thermal desalination process, and has long been applied at full-scale, due to its capacity for large production. Nevertheless, evaporation requires significant energy to overcome the latent heat (539 kcal/kg) of water (Elcock 2010), and energy consumption for vaporizing water accounts for 40~50% of the total operational cost in MSF (Miller 2003, Rajvanshi 1981, Thomas 1997). In addition, crystallization of calcium in the MSF system often causes a scaling problem, which reduces facility lifetimes. In contrast, RO is the fastest-growing desalination technology due to its high performance, convenience of operation, lower energy consumption than MSF, and flexibility in applicable scale. It does, however, need high pressure to induce osmotic pressure, and the electrical energy required for this accounts for 44% of the

^{*}Corresponding author, Professor

^a These authors contributed equally to this work.

total costs for the seawater RO (SWRO) process (Banat and Jwaied 2008). In addition to energy consumption, RO membranes are susceptible to fouling, which demands either frequent cleaning, or membrane replacement (Shannon *et al.* 2008).

ED is a membrane-based technology in which charged cations and anions can separately migrate across ionexchange membranes, by using electrical potential differences (Miller 2003). However, desalination applications of ED are limited to low TDS water (i.e., brackish water) treatment, due to its high energy consumption (Speigler and Sayed 1994). Recently, forward osmosis (FO) has gained increasing attention, due to its potentially low energy consumption, compared to SWRO (Mazlan et al. 2016). FO uses the osmotic pressure difference between the feed and draw solutions as a driving force to induce water transport across a semi-permeable membrane (Sun et al. 2017). In addition to a lower energy consumption, FO technology has other benefits, such as low fouling propensity, and ease of cleaning. Nonetheless, the mechanisms of the FO process are not yet clearly determined, and the molecular-level behavior of water and ions between the membranes still needs to be understood (Yip et al. 2010, Tiraferri et al. 2011, Qiu et al. 2012). In addition, the effective recovery of the draw solution from the FO process needs to be resolved, in order to expand its application to full-scale plants.

A desalination method using solvent extraction, referred to as a directional solvent extraction (DSE) process, has been very recently attempted (Kleinguetl, 2011, Alotaibi et al. 2017). In this method, water is separated from a saline water by using the difference in the solubility between water and a solvent, as temperature changes. The idea for this process was first proposed in the 1960s (Davidson et al. 1960). However, there has been little technical development of the relevant technologies to date. This method may have several benefits, such as flexibility of its processing scale, simplicity in process configuration, and less energy consumption, compared with other desalination processes. Its technological state is still immature and not robust, however, and its application to date has been limited to water recovery from a low salinity water of 5,000 ppm, which is much less than that of seawater (Davidson and Hood 1964). To date, only a limited number of studies has been done on this technology, due to lack of understanding of the molecular interactions among the water, the salt ions, and the solvent. More recently, Bajpayee (2011) found that some edible oils and long chained carboxylic acids could absorb water while rejecting other soluble substances, including NaCl. They described such phenomena from a thermodynamic aspect, by using free energy for the interaction of water-solvent-ions. Thereafter, many researchers have continued attempts to reveal the mechanisms for DSE through various experiments and simulations (Rish et al. 2014, Sanap et al. 2015, Chandran and Shah 2018). However, most of DSE studies done to date have used carboxylic acids such as octanoic acid and decanoic acid as a solvent. To our best knowledge, DSE using amine solvents have been little studied and reported.

The goal of this study was to investigate the potential of desalination by solvent extraction, using three non-aqueous

Table 1 Chemical properties of three selected NASs used for desalination experiment

	NAS 'A'	NAS 'B'	NAS 'C'
Chemical formula	HgC NH2	H ₃ C NH CH ₃	HgC NH2 CH3
	C8H19N	C8H19N	C8H19N
Molecular weight (g/mol)	129.25	129.25	129.25
Density (g/mL)	0.78	0.77	0.79
Boiling point (℃)	180	160	169
Solubility (g/L)	0.2	3.5	<1

solvents (NAS) with different molecular structures. The desalination performance of these three NAS was experimentally assessed, in terms of water absorption and salt rejection. In addition, the fates of water and salt in the three NAS were analyzed by using the molecular dynamic (MD) simulation method, which illustrated the interaction of the water and the salt ions with the different NAS.

2. Materials and methods

2.1 Selection of NAS

Three amine compounds were selected as the candidate NAS for DSE desalination; they had the same molecular formula, with C8 organic carbon links, with one amine group (N-H) bonded in a different position for each. This amine group (N-H) was selected, as it possessed strong hydrogen bonding, which may have allowed interactions with water or ions. The selected NASs were differentiated by their chemical structures. The N-H group was located at the end of the organic chain in NAS 'A', whereas that of NAS 'B' was located at the center of the organic chain. NAS 'C', which had the N-H group located at the end of the chain, like NAS 'A', had an ethyl group (CH₃CH₂) as a branch, in the middle of the organic chain. Table 1 shows the major properties of the three selected NAS.

The selected amines had the same molecular formula, but different properties, such as boiling point, and solubility, due to their structural difference. In particular, 'A' and 'C' showed very low solubility in water, while, 'B' was slightly higher, even though it was immiscible with water.

2.2 Desalination experiments with the selected NAS

Synthetic saline water was prepared by dissolving sodium chloride in distilled water, at 0.5 M, which is close to the concentration of seawater. 100 mL each of NAS and synthetic saline water were separately added to a 500 mL beaker, and then mixed at 300 rpm with magnetic stirring, for 10 min. After mixing, the mixtures quickly separated into their NAS and brine phases, due to differences in the densities and polarities between the NASs and water. Separation continued under quiescent conditions, for 5 min, after which the NAS (upper phase) was carefully removed. The desalination performance was then estimated in terms of water absorption and salt rejection efficiencies. The water content in the NAS was measured using Karl-Fischer analysis (KF-890, Metrohm, Switzerland).

The water absorption efficiency of a NAS can be calculated by measuring its water content increase, while the salt rejection efficiency can be obtained by measuring the before-and-after Cl⁻ ion concentration in the synthetic saline water, and can be defined as follows:

Water absorption efficiency (%)
=
$$\frac{Water \ mass \ in \ the \ solvent}{Total \ mass \ of \ solvent} \times 100$$
 (1)

Salt rejection efficiency (%)
=
$$\frac{Residual \ mass \ of \ Cl^{-}in \ the \ brine \ phase}{Initial \ mass \ of \ Cl^{-}in \ saline \ water} \times 100^{(2)}$$

Cl⁻ concentrations in the NASs and residual water (brine) were measured using ion chromatography (ICS-900, Dionex, U.S.A.).

2.3 MD simulation for desalination with solvent extraction

Molecular dynamic (MD) simulations were conducted using a Desmond molecular dynamics package (Schrödinger Release, 2018-2 Desmond). A threedimensional structure for each NAS was prepared using 2D-Sketcher, in Schrödinger suites, and was optimized using the Jaguar quantum mechanics (QM) application of the Schrödinger suite (Schrödinger Release, 2018-2 Jaguar). In the QM calculation, density functional theory (DFT) was used with a B3LYP hybrid function, and a 6-31G** basis set. Atomic partial charges of the NAS were also calculated, using the electrostatic potential (ESP) fitting method.

Desmond system builder was applied to build the initial system for MD simulation. First, 1,000 NAS molecules were randomly arranged in a three-dimensional space. Then, water molecules were also randomly arranged in a 1:1 water: NAS ratio (v:v), by using the TIP3P water model. Concentrations of Na⁺ and Cl⁻ ions were set at 0.5 M, respectively. OPLS3 force field was employed to describe all the molecules, and calculate explicit interactions among atoms. The NPT ensemble was employed in the MD simulations. To maintain a constant temperature and pressure during the MD simulations, the Nose-Hoover thermostat method was set at the reference temperature of 300 K, and the Martyna-Tobias-Klein barostat was set at the reference pressure of 1 bar. After performing a series of minimizations to relax the MD system, MD simulations were run for 100 ns.

3. Results and discussion

3.1 Experimental results of directional solvent extraction



Fig. 1 Phase separation after mixing NAS and brine: (a) NAS 'A', (b) NAS 'B', (c) NAS 'C'



Fig. 2 Comparison of water recovery and salt rejection efficiency, for the selected NASs

In order to investigate the desalination capability of NAS, we examined the volume of water transferred from 0.5 M synthetic saline water into each NAS. After mixing, the NAS and saline water mixture rapidly separated into their NAS and brine phases. The volume of NAS phase in the upper layer slightly increased, indicating that water molecules migrated to the N phase. After carefully recovering the upper NAS phase, it was observed that the volume of NASs 'B' and 'C' increased by approximately 5% and 10% of their initial volumes, respectively, while NAS 'A' increased its volume by 50% (see in Fig. 1). Water content in the solvent was more precisely measured by Karl-Fisher analysis, and water absorption efficiency in NASs 'A', 'B' and 'C' was calculated at 35.6%, 3.8% and 10.7%, respectively. The increment of water content in each NAS was not quite identical to that of the observed volume, and it was hypothesized that this was because a small NAS amount transferred to the brine phase, while the water molecules transferred to the NAS phase.

As for the water content, the Cl⁻ ion concentration in each brine phase was analyzed. The Cl⁻ concentration in the brine phase can be used as an index for the interaction of salt ions with NAS, with decreased Cl⁻ concentration in the brine phase indicating migration of salt into the NAS phase. The salt rejection efficiency for each NAS is shown in Fig. 2, in which low Cl⁻ concentration, relative to water content in the NAS, means that the NAS is capable of selectively absorbing water and rejecting salt ions, during the desalination process.



Fig. 3 Predictions for water molecules and salt ions captured inside a bilayer of NAS 'A'

In spite of exhibiting the highest water absorption efficiency, NAS 'A' showed the lowest salt rejection efficiency, at 24.7%, implying that it was unsuitable for desalination. In contrast, NAS 'B', with the lowest water absorption efficiency, showed very high salt rejection capability, at 98.9% efficiency. NAS 'C' exhibited a moderate desalination capability, in terms of water absorption and salt rejection efficiencies, with values in between those of NASs 'A' and 'B'. Considering both aspects of desalination capability, NAS 'B' was identified as the most suitable solvent among the selected NAS candidates, for DSE desalination.

The solvent 'B' exhibited a slightly lower salt rejection rate (> 99.5%) than that of carboxylic group solvent when compared to the other studies (Bajpayee *et al.* 2011, Rish *et al.* 2014). However, this solvent exhibited a better performance in terms of water absorption rate with two times higher value than that of carboxylic acids.

Unexpectedly, as shown in Fig. 1(a), when NAS 'A' was mixed with saline water, the viscosity of the mixture increased, and the mixture turned into a gel. This gelation outcome could not be clearly explained in this study, however, the formation of gelatin in a mixture of organic solvent and water has been reported previously (Trivedi *et al.* 2004, Mahapatra and Dey 2016). On the basis of these previous studies, it can be hypothesized that NAS 'A', having the hydrophilic group at the end of the carbon chain, was able to form a semi-solid structure, such as a cell membrane bilayer, which in turn formed a pocket (a kind of vesicle) where large numbers of water molecules and ions could be captured (see Fig. 3).

3.2 MD simulation for interaction of water and salts with NAS

In order to demonstrate the interaction of water and salts with NAS in DSE desalination at atomic scale, molecular dynamics (MD) simulation was carried out. MD simulation is a computer simulation method for representing the physical movements of molecules with time. MD simulations were performed for the initial states, where the NAS, water molecules, and salt ions (Na⁺ and Cl⁻) were uniformly distributed. To check the phase changes of the mixture during MD simulation, we measured the positional changes of the NAS through the root-mean-square deviation (RMSD) of atomic positions. Based on the RMSD data, all the NAS reached an equilibrium state after 5 ns and the RMSD values stabilized (Fig. 4). Stabilization of RMSD meant that NAS probably reached a steady state condition in relation to the interaction between the water molecules and the salt ions.

100 ns snapshots of MD simulations are shown in Fig. 5. NAS 'A' formed a cross-shaped cluster, surrounded by water molecule clusters. As hypothesized from the experimental results, the hydrophilic amine group (N-H) contacted the water molecules, and the hydrophobic carbon chains aggregated together, forming clusters. NAS clusters contained many water molecules inside, and the water molecules might have bonded with each other through their hydrogen bonding network. Based on the experimental results, NAS 'A' exhibited the highest water absorption capacity and lowest salt rejection efficiency, which meant no selective absorption of water from saline water. However, in the MD simulation, no NaCl ions were found inside the solvent cluster. In order to see the clusters at a macro-scale view, we extended the boundary of the MD system scale to a 3 x 3 system, by making replicas of the cluster spaces. In the extended system, the solvent clusters formed a lattice structure which surrounded many pockets (vesicles), that could contain water molecules and NaCl ions together (Fig. 6(a)). Interestingly, the lattice structure of the solvent cluster looked very similar to the general gel structure (Fig. 6(b)), which supported the observation of gelatin formation during the experiment.

In contrast to NAS 'A', NASs 'B' and 'C' formed planar-shape cluster structures (Fig. 5(b), (c)). Although neither solvent formed vesicles within the solvent clusters, small numbers of water molecules were found to migrate inside the clusters. The water absorption efficiency could be analyzed by counting the number of water molecules which migrated to the solvent clusters, and the results were 1.29% and 2.13%, for NASs 'B' and 'C', respectively. There was a significant difference in water absorption efficiency between the experimental results and the MD simulation, probably due to limitations in the scale of the MD simulation. As well, in spite of exhibiting a similarly shaped cluster, NAS 'C' showed greater water absorption capacity, due to the presence of its ethyl branch. The branch-type structured NAS 'C' looked like forming a rougher surface of clusters than the straight chain structure of NAS 'B', and the increased interfacial surface area, between water and NAS 'C' clusters, may have led to greater opportunities for transferring water and salt ions into the solvents.

In contrast the experimental results, the MD simulation did not demonstrate the movement of both Na^+ and Cl^- to inside of the solvent clusters. In a larger scale view shown in Fig 6 (a), it appeared that some of these salt ions are placed at the boundary of solvent and water. Nevertheless, different from water molecules, the fate of salt ions were not clearly demonstrated by MD simulation in this study. Thus, it is necessary to more clarify the interaction of salt ions and solvent through modifying the MD simulation in the future.



Fig. 4 RMSD results of MD simulations



Fig. 5 MD simulation results for DSE desalination using (a) NAS 'A', (b) NAS 'B', (c) NAS 'C'. The red and white symbols represent water molecules, green and blue symbol are Cl⁻ and Na⁺ ions, respectively, and the vacant space is the solvent layer



Fig. 6 Comparison of the lattice-structure of (a) NAS 'A', and (b) general structure of gelatin (reproduced from Sakai T., 2013). The red and green symbols represent salt ions, and the gray layer is the solvent

4. Conclusions

DSE desalination is a concept that is completely different to current technical approaches, but it can be a cost-effective and flexible technology for desalination, even though its technical state is not yet robust. Three amine group solvents, with the same molecular formula but different molecular structures, were selected as NAS for testing. The different NAS structures led to different desalination performance. NAS 'B', with its amine group in the center of a straight C8 chain, showed the best desalination performance, in terms of both water absorption and salt rejection efficiency. NAS 'A' exhibited substantially higher water absorption efficiency but the lowest salt rejection efficiency, due to formation of water pockets (vesicles) inside the solvent clusters, and demonstrated by MD simulation. The MD simulation results matched relatively well with the experimental

results, even though the process needs to be modified by expansion of the scale, and diversification of the conditions used for the simulations.

The mechanisms involved in the interaction of water and salt ions with NAS, in DSE desalination, still remain poorly understood. In the future, experiments supported by MD simulation will be helpful for better understanding the mechanisms of DSE desalination, and for enhancing its performance.

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