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Chapter

Short Review of Salt Recovery from Reverse Osmosis Rejects

Boopathy Ramasamy

Abstract

The membrane treatment is a physical separation which also generates considerable amount of waste, called as reject/concentrate. The reject/concentrate is more than three times concentrated than the feed water in terms of feed water salts. Recovery of valuables from reverse osmosis (RO) reject for its reuse of inorganic salts would be most obvious solution to eliminate environmental damage. In this report what are the available methods for the recovery of valuables from waste saline stream by selective crystallization method, chemical precipitation and physico-thermal route discussed in details. Also, methods to treat organic contamination in the residual solution through advanced oxidation treatment methods.

Keywords: reverse osmosis, salt recovery, reactive precipitation, crystallization

1. Introduction

The demand of potable water led to significant improvements in the membrane separation process in water and waste-water treatment. Especially for developing countries, the demand of water supply is increasing and no less provision to mitigate the challenges. The state and central governments agencies are formulating strategy through environmental policies to mitigate the water scarcity. During last three decades significant expansion and upgradation of membrane filtration have been happening around the globe to lower it production costs. In market, there are many custom made membrane technologies that are available for both domestic and industrial applications. Reverse osmosis is one of the important technologies among which has energy and cost effective membrane system for producing potable water from brackish and saline water sources [1–7]. RO process can generates 50–80% of water drinkable or dischargeable quality as permeate [7] and the balance 20–30% formed as RO reject or retentate or concentrate. Generally, concentrate is allowed to avoid membrane fouling, coagulation and flocculation process over membrane surfaces as it cannot be further purified due to high osmotic pressure. The common application of membrane separation processes comprising ultrafiltration and reverse osmosis for the disposal of reverse osmosis (RO) rejects through thermal evaporator or multiple effect evaporator.

1.1 Characteristics of membrane concentrate

Membrane separation is a physical process which involving the separation of particulate, and dissolved organic/inorganic compounds from a feed liquid using a semi-permeable membrane system. The feed stream is fractionated into two streams:
(i) a permeate stream that contains the solvent (water) that passes through the membrane, and (ii) the reject stream known as concentrate, reject or brine contains the solute. The characteristics of this reject stream depend primarily on the membrane technology used, the quality of feed water, the percent recovery of water, the physico-chemical treatment methods followed for cleaning procedures. The volume of reject stream varies widely from 10 to 60% of the feed water volume [7]. However, the reject stream arises from industrial effluents such as textile, pharmaceutical and tanneries are turbid and opaque, may be due to the presence of micro and macro organic molecules and inorganic ions, such as chlorides, sulfates, phosphate, carbonate, bicarbonate, sodium, calcium, magnesium and other heavy metal ions [8]. The characteristics of RO stream generated in leather industry are presented in Table 1. The TDS of water is increased from 5.58 \pm 1.8 to 20 \pm 6.14 g/L in RO reject stream leaving product water with a TDS of 0.25 \pm 0.13 g/L during separation process. The TDS concentration of RO rejects four fold higher than the initial TDS concentration. Similarly, concentration of sodium, chloride and sulfates ions were found to be increased significantly after RO separation process in reject stream.

1.2 Impacts of membrane concentrate discharge without treatment

Discharge of RO reject stream into sea/ocean is considered to cause the “sea desert” in vicinity to pipe outlet because of dissolved substance with high specific weight and thus sink in bottom of the sea/ocean, which severely affecting local marine biota. Marine species have been affected by the salinity of the brine discharged into the sea include grass prairies called such as Cymodocea nodosa and Caulerpa prolifera or red algae [9]. Direct land disposal of RO reject stream from effluent treatment plants caused soil and groundwater contamination by the diffusion of inorganic impurities from it, and thus soil and ground water are turned unsuitable for human consumption for their harmful or toxic substances. Hence, there has been constant exploration to manage the RO reject stream.

1.3 Options for membrane concentrate disposal

Various options have been reported for the disposal of RO reject stream generated in membrane separation. This option is discharge to surface water, deep wells,
solar evaporation ponds, and thermal evaporators. The selection of technologies for the disposal of RO rejects is based on the prevailing environmental regulations, investment and maintenance costs, and site-specific conditions [10, 11].

The reduction of concentrate volume is being achieved by incorporating zero liquid discharge technologies. The concentrate streams after conventional RO are being evaporated in thermal evaporators to sufficiently dry inorganic salt. The most common way is vertical falling film brine concentrator followed forced-circulation crystallizer, where it is heated above its normal boiling temperature with steam through heat exchanger. It requires 65–80 kWh of power per 1000 l of crystallizer feed water. Crystallizers and spray dryers have been implemented at the commercial level to reduce RO reject stream into a solid product for landfill disposal. One or more evaporation steps could also be considered to recover small amounts of water from the most soluble salts in RO reject stream. The primary practical obstacle in implementing thermal evaporators is size and complexity of the equipment. In addition, evaporators and crystallizers are relatively complex to operate and high energy intensive process compared with other zero liquid discharge (ZLD) methods.

The evaporators and crystallizers are used to reduce the reject volume up to 5% of the feed volume and the rest 95% was reclaimed as distillate (water) after condensation. Generally, the reject stream generated from leather industry is evaporated in solar evaporation pans to reduce its volume (for small volume of discharge and making use of solar energy to reduce the cost of evaporation) or in a multiple effect evaporator (MEE) (for large volume of discharge) leaving behind a solid residue known as residue after evaporation (RAE).

1.4 Limitations on disposal of residue after evaporation generated from leather industry

The disposal of RAE onto secured landfill sites is banned by the pollution control agencies because the constituent ions are suspected to be leached into aqueous solution, and the treatability of leachate would be more difficult for its high salinity. Hence, RAE is collected and being stored in the storage yard without further reusable options in many textile and leather industries. The high concentration of mixture of inorganic and organic salts present in RAE restraints it from disposal [13, 14].

2. Desalting process for RO concentrate

Recovery of salts from RO concentrate is increasing for its environmental safe way to reuse of waste volume of reject. The change of solution temperature either by evaporation and cooling used to extract salts from concentrate has been widely used worldwide. In which, electro-dialysis, ion-exchange, eutectic freezing, and chemical processing are being practiced to recover salts from concentrate. Further, in advance hybrid systems which consisting of combination of two or more separation process techniques, such as the nanofiltration—reverse osmosis—thermal processes, are being deployed actively to increase the amount of extracted salt and reduce the final volume of reject.

Buckley et al. has proposed detailed routs for the management of RO reject [12]:

i. Use of engineering knowledge: incorporation of engineering unit operation/process to reduce dissolved solids in the reject stream.
ii. Chemical conversion of reject to products: chemical conversion of rejects to other reusable salts from waste RO reject.

iii. Direct and indirect discharge of concentrated brine by dilution without affecting receiving environment.

iv. Stabilization of concentrate to inert material: stabilizing the waste concentrate into chemically stable material.

2.1 Evaporation and cooling

Recovery of valuable salts or minerals can be obtained from RO concentrate or brackish water by altering solution temperature either by evaporation or cooling effect. There are reports on evaporators are being practiced in brine management, among which multiple effect evaporator (MEE) are most promising and cost effective. The MEE operate based on the principle of reducing the vapor pressure of solution within the system to permit boiling occurs at low temperature. The multiple effect evaporator feed water is boiled and pumped into tube side in the evaporator in series. The outcome of steam has been condensed over the tube wall of the evaporator and collected as water to reuse. The excess heat is further used for the boiling of inlet water.

In other way, vapor compression distillation (VCD) is used for the desalination process. In VCD inlet water is boiled through heater to vaporize and discharged through evaporative compressor. The generated vapor has been compressed and used as steam supply for boiling concentrate and the condensate product is obtained after compression process.

2.2 Electro-dialysis (ED) and ion exchange

Electro-dialysis system consists of anion-exchange and cation-exchange membranes are being arranged alternately in a large cell of compartment between an anode and a cathode. The influence of applied electric field, the various ions could migrate towards the electrodes based on its ionic charge. The membranes are permeable only to cations or anions, through which the water between the membranes are alternately depleted and enriched with salt ions. The cation membranes allow only positively charged ions to diffuse through them. Similarly, anion exchange membrane allows only negatively charge ions. Electro-dialysis is being considered used as a pre-treatment or a pre-concentration method for brine management [15–17]. This method of application significantly reduces the concentration of calcium or sulfate ions from gypsum crystallization during further evaporation [15]. However, fouling by colloidal material, organics, and bio-growth should be taken care for the effective and sustainable use of the equipment.

The performance of ion exchange processes based on packed bed column resin, which are generally organic resins that contains hydrogen ions and is capable of exchanging positive ions present in the feed water. The ion exchange processes are being studied, investigated, and applied for many desalting process over several decades [18–25]. However, this method of treatment applies only to low concentrations of salts containing brine water and its cost of regeneration is also higher.

2.3 Eutectic freezing crystallization (EFC)

In this process, the feed concentrate stream is frozen continuously until it reaches a eutectic temperature. The ice being forming.
If the unsaturated concentrate is reaching its freezing point. At the specific eutectic point of crystallization, the brine salt is crystallized out as product. The energy required for the EFC process are found to be very less than the conventional method of evaporative and cooling crystallization process and its theoretically possible way to complete the conversion of concentrate water into water and solidified solutes. Through this route magnesium sulfate heptahydrate (MgSO$_4\cdot7$H$_2$O) from a magnesium sulfate industrial stream is being recovered using EFC process [26, 27]. In which MgSO$_4\cdot12$H$_2$O was formed in the crystallizer and after recrystallization MgSO$_4\cdot7$H$_2$O is formed spontaneously. In addition to EFC, coupling of cooled disk column crystallizer (CDCC) helps to recover CuSO$_4$ crystals from copper sulfate solution [28, 29]. The cost towards energy required for EFC can be reduced up to 70% than conventional evaporative crystallization processes, further 100% conversion of concentrate into water and salt separation is achieved by this route.

2.4 Chemical process

Recovery of CaCO$_3$ from nanofiltration reject is being achieved by reactive precipitation on addition of NaHCO$_3$/Na$_2$CO$_3$ aqueous solution [30]. Similarly, recovery of MgSO$_4\cdot7$H$_2$O from the reject from seawater nanofiltration, Ca$^{2+}$ ions were precipitated as carbonates by reaction with NaHCO$_3$/Na$_2$CO$_3$ to get calcium sulfate by precipitation, Sodium-bi-carbonate solutions are being produced by reactive transfer of carbon dioxide into sodium hydroxide solutions. This technique has been used successfully to recover magnesium sulfate from sulfate rich brine, rock forming minerals, and salty lake water [31–33]. There are many sequential extraction of salts from rejected brine thus high concentration of dissolved sulfate, potassium, and magnesium salts are being separated through multiple effect evaporator and cooling crystallization method, reactive precipitation methods [15, 28]. The use of lime favors selective separation of magnesium hydroxide from concentrate solution and thermal calcination of concentrate having sodium sulfate helps to recover Na$_2$SO$_4$ from wastewater [34]. The reject generated from textile dyeing industry contains large amount of Na$_2$SO$_4$ and thus being recovered through multiple effect evaporation and/or calcinating the concentrated reject in a muffle furnace at a temperature range of 300 and 800°C [35].

2.5 Extraction of potentially profitable material from RO rejects

2.5.1 Calcium

The recovery of calcium from RO reject is done to avoid secondary RO scaling [36–38]. Bond and Veerapaneni [36] have developed detailed methodology for the recovery of calcium carbonate by chemical precipitation, in particular to separate calcium during desalination. Several other researchers have evaluated fluidized bed crystallizers for the production of calcium carbonate pellets from RO reject [36, 39], further studies have been reported on influence of anti-scalants, impurities, metals, and ions on calcium carbonate precipitation [40–42]. The concentration of calcium ions in seawater and desalination reject is relatively high, through this calcium carbonate pellets has been prepared from brackish water in Southern California [39]. However, the extraction of calcium sulfate from RO reject has not received significant attention, due to low price of commercial grade gypsum. Also, the mechanisms of calcium sulfate precipitation have been observed to form scaling minimized equipment failure in separation process [43–46]. The ion-exchange resin is being used to selectively extract calcium sulfate salts from RO reject by controlling the pH of the mixture of anion and cation regeneration solution.
2.5.2 Magnesium

The main unit processes used for the extraction of magnesium is evaporation cum crystallization, precipitation, and ion-exchange. Ohya et al. [47–49] proposed a series of integrated processes (crystallization, electro-dialysis, ion-exchange) to recover salts of calcium carbonate, sodium chloride and magnesium sulfate from RO reject. Drioli et al. identified a process of membrane crystallization/distillation to separate out various inorganic salts from reject streams from an integrated NF/RO process [30, 50]. In Russia, a large scale extraction plant is also being operated for the recovery of magnesium from seawater using ion-exchange, and extraction of magnesium from RO reject from seawater desalination system.

2.5.3 Potassium

Worldwide potash consumption is increasing every year approximately at a rate of 3% due to population growth and other increased demand for fertilizers [51]. The main source of potash production is done by conventional shaft mining or deep-well solution mining process techniques. Currently, potassium is being produced from seawater as a byproduct from solar salt evaporation. As an alternative to solar evaporation/precipitation, several researchers have suggested that, potassium could be produced from RO reject using an evaporation/crystallization process [52, 53]. The extraction of potassium is done using natural zeolite (clinoptilolites) ion-exchange materials which has high exchange capacity for potassium through a two-step dual-temperature process.

2.5.4 Sodium

The production of sodium compounds from desalination reject is obtained through evaporation technologies, followed by crystallization [54, 55], membrane crystallization [56, 57], electrodialysis followed by multiple effect distillation (ED/MED) [54–58], and evaporation ponds [59, 60]. Membrane crystallization (MCr) is being practiced to produce relatively pure salt crystals from a synthetic NF reject solution having calcium and magnesium [66]. Tanaka et al. [17] developed an electro dialysis process for the production of salt from seawater reverse osmosis (SWRO) reject with less than 80% energy than conventional process. A similar process developed by Davis [47] on electrodialysis metathesis which has integrated evaporator unit to separate out sodium sulfate and sodium chloride [61]. The SAL-PROC process (Geo-Processors USA Inc) is being used to produce sodium chloride, calcium sulfate, calcium chloride, and magnesium hydroxide from concentrated solutions including brackish water reverse osmosis (BWRO) and seawater reverse osmosis (SWRO) concentrate.

2.5.5 Nitrogen

In general, RO reject stream was found to be more than 40 mg of nitrogen per liter. The available method to recover ammonia-nitrogen by struvite precipitation, since extraction of ammonia is economically poor.

2.5.6 Sodium chloride

The SWRO reject through either electro-dialysis (ED) or electro-dialysis reversal (EDR) step is sufficient to separate out impurities and that the salt produced is fit for human consumption; however, there is little information available on final
product purity using this approach Tanaka et al. [17]. Electrolytic method of simultaneous separation of chlorine and sodium chloride has good market potential for the effective management of RO reject. Melian-Martel et al. [62] used membrane electrolytic cells to recover chlorine, hydrogen and sodium hydroxide from seawater RO reject. Boopathy et al. reported separation of sodium chloride from the RO reject generated in leather processing industries through reactive precipitation techniques [63].

2.5.6.1 Precipitation of sodium chloride from evaporated residue of RO rejects

The movement of ions during precipitation is expressed in the form of chemical equations as given below:

\[
\text{RAE} + \text{H}_2\text{O} \rightarrow \text{Na}^+, \text{Cl}^-, \text{Ca}^{2+}, \text{SO}_4^{2-}, \text{Mg}^{2+}, \text{Org}^- / \text{Org}^+ \text{, H}_2\text{O} (1)
\]

\[
(\text{Na}^+, \text{Cl}^-, \text{Ca}^{2+}, \text{SO}_4^{2-}, \text{Mg}^{2+}, \text{Org}^- / \text{Org}^+, \text{H}_2\text{O}) + \text{HCl(g)} \rightarrow \text{NaCl(s)} + [\text{H}_3\text{O}^+] + \text{Na}^+ + \text{Cl}^- + \text{Ca}^{2+}, \text{SO}_4^{2-}, \text{Mg}^{2+}, \text{Org}^- / \text{Org}^+ (2)
\]

The residue of RO rejects has been dissolved in water to prepare saturated RAE solution as shown in Eq. (1). The increase in ionic concentration in the saturated solution shifts the reaction to backward direction by common ion effect. In this study, hydrogen chloride gas was prepared and purged to increase the concentration of Cl\(^-\) ions in the RAE solution. The incremental increase in Cl\(^-\) ion concentration shifted dynamic equilibrium by increasing the ionic product of Na\(^+\) and Cl\(^-\). The ionic product of Na\(^+\) and Cl\(^-\) exceeded the solubility product of sodium chloride \(\text{[solubility product of NaCl, (K}_{\text{sp}}\text{) is 36 (mol/L)²]}\) and thus the precipitation of sodium chloride was achieved from the saturated solution of RAE as illustrated in Eq. (2). The schematic flow diagram of separation of sodium chloride from RAE solution generated in leather industry has been illustrated in Figure 1. First saturated RAE solution has been prepared by dissolving 60\% (w/v) RAE in water and the insoluble grits are removed after gravitational settlement. The clear supernatant solution was taken in reactive precipitation reactor and HCl gas has been purged continuously for the reactive precipitation of sodium chloride. The required HCl is being prepared and used spontaneously. Since the prepared HCl gas cannot be stored, if we store which may condensate and turn into liquid form. After successful purging of HCl gas the sodium chloride salt is separated out from the solution by reactive precipitation as per the reaction given in Eq. (2).

2.5.6.2 Effect of HCl gas injection time and RAE concentration on NaCl recovery

The HCl gas purging time for the separation of sodium chloride from RAE solution was carried out by varying time from 0.5 to 3 min at its native pH, 8.0 and temperature, 40°C. The optimum condition for the recovery of NaCl is achieved within 3 min of contact time as shown in Figure 2a. The optimum time of 3 min of contact time yield 81% recovery of sodium chloride. This is explained that the equilibrium was established i.e. the rate of precipitation of NaCl becomes equal to the rate of dissolution of NaCl in the solution. The mass of precipitated NaCl at the optimum time was 26.7 g with 81% recovery with respect to the dissolved salt concentration (solubility of NaCl is 35 g in 100 mL of water).

The concentration of RAE [40–65\% (w/v)] was varied to identify the effect on precipitation of NaCl. The results in Figure 2b, shows that the percentage of salt recovery increased with the increase in concentration of RAE. In general, precipitation depends on the concentration of dissolved ions in solution. As the initial
concentration of RAE increased, the dissolved ions concentration was also increased in the solution and reached the saturation limit at concentration 60% (w/v). The maximum amount of NaCl precipitation was achieved with 82% recovery for 60% (w/v) RAE solution. Further to evidence that the recovered salt is NaCl, SEM and EDAX analyses were carried for the recovered NaCl as shown in Figure 3. The surface morphology of RO reject looks aggregated mass like structure and thus may be due to mixture of many inorganic and organic salts. This is confirmed by EDAX spectrum shows presence of inorganic salts. However the recovered salt has a cubical structure, which is a characteristic morphology of sodium chloride and thus it claim that the recovered salt is sodium chloride. Further EDAX spectrum peak observed only for Na and Cl and thus confirmed that the recovered salt is NaCl.

2.5.6.3 Mass balance on preparation of saturated RAE solution

\[
\text{mass of RAE + deionised water} \rightarrow \text{saturated RAE solution + grit} \\
0.6 \text{ kg} + 1 \text{ kg} \rightarrow 1.32 \text{ kg} + 0.28 \text{ kg} 
\] (3)

The mass of saturated solution of RAE was 1.32 kg, obtained by dissolving 0.6 kg of RAE in 1 l of deionized water. The undissolved grit (0.28 kg) mainly consists of sand, lime and clay being non-hazardous in nature, which can be disposed off onto secure landfill.

2.5.6.4 Mass balance on precipitation of sodium chloride

\[
\text{saturated RAE solution + HCl gas} \rightarrow \text{precipitated sodium chloride} \quad + \quad \text{residual solution} \\
1.32 \text{ kg} + 0.105 \text{ kg} \rightarrow 0.268 \text{ kg} + 1.157 \text{ kg} 
\] (4)

The maximum precipitation of sodium chloride of 0.268 kg was resulted from 1 l of saturated RAE solution under the optimized conditions.
The proposed process for the management of RAE was relatively lower in cost than the other disposal methods, and also the process has the scope to recover sodium chloride. The proposed process recovered 0.203 kg of NaCl from 1 kg of RAE. The resulted acidified supernatant solution (RAS) was considered for separation of sulfate ions as calcium sulfate. The sulfate ion in the RAS solution and synthetic RAS solutions were separated by the addition of various neutralizing agents. Figure 2 illustrates the precipitation of sodium chloride (a) effect of time (conditions: pH, 8.0; temperature, 40°C; mass of RAE, 60% (w/v)), (b) effect of concentration of RAE (conditions: time, 3 min; pH, 8.0; temperature, 40°C).

The proposed process for the management of RAE was relatively lower in cost than the other disposal methods, and also the process has the scope to recover sodium chloride. The proposed process recovered 0.203 kg of NaCl from 1 kg of RAE. The resulted acidified supernatant solution (RAS) was considered for separation of sulfate ions as calcium sulfate. The sulfate ion in the RAS solution and synthetic RAS solutions were separated by the addition of various neutralizing agents.
agents. Among the selected neutralizing agents, Ca(OH)$_2$ was effective for the separation of sulfate ions from the RAS solution and synthetic RAS solutions. The total cost for the management of 1 kg of RAE by the proposed process was 0.155 USD while the cost on landfill disposal was 0.11 USD. The recovered salts proposed to be reused for the hide/skin presentation in slaughter house [63].

### 2.6 Integrated process

Salt recovery or recovery of valuables from concentrate are being effectively done through an integrated approach by combining one or more separation process which serve as a pretreatment or post treatment step [64]. In electro-dialysis for concentrating reject brine solution, multi-stage flash being employed for desalting water, and crystallization for recovering salts, or with RO may be used instead of the multistage flash evaporator [65]. In another approach, RO reject are being supplied to an ion-exchange membrane electrodialyzer as post treatment, and then concentrated brine from the electrodialyzer is sent to a multi-effect vacuum evaporator to crystallize the salts present in water stream [58]. Variety of inorganic salts is also sequential extracted from rejected brine for the high concentration levels of dissolved sulfate, potassium, and magnesium salts through multiple effect evaporation and cooling of saline wastewater, chemical reactions, crystallization, washing, and dewatering. The processes developed by researchers to recover various valuables from RO reject are presented in Table 2. The combined process helps to recover both salts and water from textile rejects [66–68].

Figure 3. Scanning electron microscopy images of: (a) RAE, (b) recovered NaCl from RAE and energy dispersive X-ray spectra of (c) RAE, (d) recovered NaCl from RAE.
3. Conclusions

The thermal routes of evaporation are the most studied process techniques for the recovery of inorganic salt from RO reject; however, membrane separation techniques are cheaper for the recovery of product quality. The membrane separation process has generates reject volume which need further treatment. The ion-exchange, electro dialysis, eutectic freezing, and chemical reaction are also being explored more in recent times for its cost and efficiency on recovery of valuables from the reject stream. The integrated systems are being studied by combining one or more unit operation or process techniques to increase the recovery percentage of valuables. The selection of process or techniques may be selected based on the salt to be recovered, geological, hydrological, climatic, and economic conditions for its local specific.

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Conflict of interest

There is no conflict of interest with any funding agencies.

Nomenclature

| Sr.No | Processes | Recovery of valuables |
|-------|-----------|-----------------------|
| 1     | EDR, precipitation, evaporation | Br₂, Mg(OH)₂, NaCl |
| 2     | ED/EDR, MSF, crystallization | NaCl |
| 3     | ED/EDR, MED, crystallization | NaCl |
| 4     | ED/EDR, MSF, MVC, precipitation, NF, Mer | NaCl, Mg, Cl₂, NaOH, CaCO₃ | Kim, 2011 |
| 5     | SAL-PROC process, precipitation, evaporation | CaSO₄, Mg(OH)₂, NaCl, Na₂SO₄, CaCO₃ | Ahmed et al., 2003 |

Table 2. Recovery of valuables from RO reject through integrated approach.
MCr       membrane crystallization
EDM       electrodialysis metathesis
BWRO      brackish water reverse osmosis
SWRO      seawater reverse osmosis
EDR       electro-dialysis reversal
RAS       resulted acidified solution
USD       US dollar

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