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Pretreatment Process on Reverse Osmosis Brine as Electrodialysis Feed

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Abstract. Reverse Osmosis (RO) Brine is waste generated from the desalination process using the RO method. RO Brine is generally directly thrown back into the sea, even though it has the potential to be reprocessed because it still contains a variety of ions in it. The best method in RO Brine processing is Electrodialysis. But it has a problem of decreased membrane performance caused by the formation of fouling. The fouling problem can be overcome by doing a pretreatment process to eliminate impurities contained in RO Brine, one of which is Ca^{2+}. The existence of Ca^{2+} can trigger the formation of $CaSO_4$ deposits. Therefore, it needs excess reagent Na2CO3 with a certain amount to eliminate the whole Ca^{2+}. Currently, it isn't yet known the best pretreatment conditions that can eliminate impurities ions and produce high concentrations of NaCl. Pretreatment trials are needed in various variations of reagents amount to reduce impurities. The purpose of this study is to find out the best RO Brine pretreatment process that will later be used for the electrodialysis process to produce high NaCl recovery. The best results were obtained in the pretreatment process with variations NaOH excesses by 15% and Na_2CO_3 by 30% from the ideal stoichiometry.

Introduction

The process of desalination of water in the power plant industry uses many Reverse Osmosis (RO) methods [1]. Seawater is processed to produce freshwater that will later be used as boiler feedwater [2]. But 30% of the processed RO becomes freshwater while the remaining 70% is wastewater (water that still contains some ions in it) that is not used and discharged into the sea. The ions contained in such wastewater are Cl^-, Ca^{2+}, Mg^{2+}, K^+, Na^+ [3]. The disposal of RO waste into the sea is a very unfortunate action because if processed, the RO Brine can produce industrial salt (NaCl) [4].

RO brine processing can be done by several methods that are broadly divided into membrane and thermal technology [5]. Thermal technology is a method by which saltwater is heated and then produced water vapor that will be condensed into clean water. This process can be done without the need for a pretreatment process and can eliminate existing biological impurities, but this process requires high energy, expensive tool manufacturing costs as well as serious corrosion, scaling, and fouling problems [6]. While membrane technology is the process by which saltwater will be separated into two phases, then there will only be one phase that can pass through the membrane. The phase shift is affected by the driving force, which occurs due to differences in pressure, concentration, temperature, and potential differences [7]. In its application, membrane processes require a pretreatment process and cannot eliminate biological impurities, but membrane processes require less energy and the cost of making tools is not too high when compared to the thermal process [8]. Compared to some other methods for processing RO Brine, processing methods utilizing membrane technology are often used and developed [9].

One membrane technology that can be used is electrodialysis (ED) [6]. ED was first developed in the 1950s. The function is to demineralize brackish water. Nowadays ED has been applicated in many functions, for example, to reduce inorganic substances in water, in-home water treatment and water recovery for reverse osmosis reject [10]. Electrodialysis (ED) is a method to reduce ion or salt that is contained in solution by conducting electric current to the process. There are two different types of electrodes (cathode and anode) that will be used to transfer the electric current and each of it has a different electrical pole [11]. In between anode and cathode, there are stacks of specific membrane for the transfer of positive and negative ions [12]. Electrodialysis methods can eliminate TDS up to 50-99% [13]. It has lower energy consumption [14] and higher membrane life [6][15]. However, in membrane technologies such as ED, major types of IEMs fouling that are commonly found are: biofouling and colloidal [16][17]. Membrane biofouling is made up by the attachment of microorganisms which are mainly bacteria to the membrane surface, followed by their growth and also multiplication, in this way it can eventually begin to form a biofilm on the membrane surface [18]. The electrical efficiency used to decrease over time caused by the formation of deposits on one side of the membrane (fouling). Deposits on the membrane (fouling) will decrease the limiting current, causing a decrease in electrodialysis performance. Fouling can be caused by a buildup of neutral or colloidal material resulting from ion exchange [19]. With reduced fouling in the membrane, the performance of the membrane will become lighter so that it will reduce the energy consumption used and can obtain higher NaCl purity [13]. To prevent fouling, an effective pretreatment process is needed in the solution that will be used as a feed in the electrodialysis process to remove ion impurities.

Materials and Methods

Materials. RO Brine is obtained from Paiton Power Plant, East Java, Indonesia. Reagents for the pretreatment process consist of sodium bicarbonate (Na_2CO_3) Pro Analyst, sodium hydroxide (NaOH) Pro Analyst, barium chloride ($BaCl_2$) Pro Analyst, Poly Aluminum Chloride (PAC) 6%, flocculant accofloc type A310, filter paper Whatman which sized 1 μm, conventional filter paper and HCl solution 0.1 M.

Methods. Before pretreatment, the ion content in the solution RO Brine is analyzed to find out the amounts of reagents that must be added to the feed (RO Brine). The pretreatment process consists of 3 stages [20]. The first stage is the addition of NaOH solution with a variation of excesses of 0% in the feed until the formation of deposits $Mg(OH)_2$ and Na_2CO_3 with stirring for 1 hour. Then the results of the first stage are filtered with Whatman filter paper 1 μm. The second stage of pretreatment is the addition of a solution of Na_2CO_3 with an excess amount of 0% on the feed in a temperature condition of 50°C to precipitate Ca^{2+} ions into $CaCO_3$ deposits with stirring for 1 hour. PAC is added slowly as a coagulant on a feed that has gone through the second pretreatment stage with stirring for 15 minutes. The precipitation process is carried out for 30 minutes until the sediment collects below. Then the solution of the results of the second stage of pretreatment is filtered with conventional filter paper. The third pretreatment stage is the addition of $BaCl_2$ solution with the amount according to the calculation of stoichiometry on the feed that has gone through the second pretreatment stage with stirring for 50 minutes until $BaSO_4$ deposits are formed. Once the precipitate is formed, the solution is filtered using conventional filter paper. Then do a pH check on the solution of pretreatment results. If obtained a pH value of >7, it is necessary to add HCl to the solution of pretreatment results so that the pH of the solution becomes 5. For the addition of NaOH and Na_2CO_3 reagents, variations were made with excesses of 5% and 25% for NaOH and excesses of 20% and 30% for Na_2CO_3 solutions. This variation aims to determine the amount of the most effective reagent that must be added to the pretreatment process in order to produce the least impurities. For every reagent, the excesses were calculated from the ideal stoichiometry. At the end of the pretreatment process, an analysis of the content of ion impurities in the solution of pretreatment results and these results are compared to all variables to determine the best pretreatment conditions.

Results and Discussion

Analysis of RO Brine's ion composition. The composition of impurities of RO Brine as feed pretreatment process is analyzed by several methods [21] [22]. Some of the impurities ions that have large amounts on RO Brine are Ca^{2+}, Mg^{2+}, Na^{2+}, Cl^{-}, SO_4^{2-} and K^{+}. The results of the analysis of the ion content are described in Table 1. It can be seen that the largest composition of RO Brine is Na^{+} and Cl^{-} ions where both ions will later be recovered by electrodialysis process to produce a solution with high NaCl content as an industrial salt feed. The results of the analysis of the RO Brine solution to be used have a lower composition of ion impurities compared to other authors' reports [23].

Table 1. Composition of RO Brine

Component	Composition [ppm]	Method of Analysis
Ca^{2+}	116,67	Complexometric
Mg^{2+}	417,56	Complexometric
Na^{+}	8725	Flame Photometry
Cl^{-}	27550	Argentometry
SO_4^{2-}	348,49	Spectrophotometry
K^{+}	132,78	Flame Photometry
CO_3^{2-}	0,6	Alkalimetry
TDS	38500	Gravimetry

Electrodialysis is a process of separation using a membrane [24]. In general, membrane electrodialysis has certain specifications and restrictions for each ion impurities as described in Table 2. The purpose of the pretreatment process is to eliminate the ion impurities content to close to the requirements of the main composition of membrane electrodialysis cells [20]. From Tables 1 and 2, it can be seen that the ion content of Ca^{2+} and Mg^{2+} contained in RO brine not met the requirement. So, it needs to be processed by pretreatment with several variables to reduce impurities by the addition of NaOH and Na_2CO_3 reagents.

Table 2. Main Requirements Composition of Membrane Electrolysis

Ca^{2+}	Mg^{2+}	Na^{+}	Cl^{-}	SO_4^{2-}
$Ca^{2+} + Mg^{2+} < 20$ ppb		118 g/L	182 g/L	8 g/L

(Casas et.al, 2012, [23])

The influence of reagent amount for impurities degradation. The pretreatment process is carried out with three stages of the process. In the first stage, NaOH reagents will be added to remove CO_3^{2-} and Mg^{2+} impurities [25]. Then the second stage is the addition of Na_2CO_3 reagents to remove Ca^{2+} impurities [25]. And the third stage is the addition of $BaCl_2$ reagents to remove SO_4^{2-} impurities [20]. Variations in the addition of reagent excesses are carried out in the first and second stages of pretreatment, namely in the addition of NaOH solution and Na_2CO_3 solution. For NaOH solutions, excesses vary by 0% (without excesses), 15%, and 25%. As for the solution Na_2CO_3 carried out excess variations of 0% (without excesses), 20%, and 30%. In previous research data [20] found that to eliminate calcium content, it takes the addition of excess reagent Na_2CO_3 by 20-30% above stoichiometric calculations. The effect of adding excesses of NaOH and Na_2CO_3 reagents to ion impurities is shown in Figures 1, 2, and 3.

The addition of NaOH to the pretreatment process aims to remove calcium carbonate solids and to neutralize carbonic acid. The addition of this reagent is also to eliminate the impurities of $Mg(OH)_2$. The reactions that occur are as follows:

$$CO_3^{2-} + NaOH \rightarrow Na_2CO_3 + 2OH^- \quad (1)$$

$$Mg^{2+} + 2NaOH \rightarrow Mg(OH)_2 + 2Na^+ \quad (2)$$

This addition can be used to handle RO Brine that has a high carbon and low calcium. In addition, the addition of NaOH also aims to increase the pH of the solution to achieve the formation of Ca^{2+} solids when the solution is added with the reagent Na_2CO_3 [20]. For NaOH reagents, the analysis's results of the first stage pretreatment can be seen in Table 3.

Table 3. Effect of NaOH Reagent Addition on Mg^{2+} Ion Reduction

Content of Mg^{2+} [ppm]			
RO Brine	NaOH Excesses 0%	NaOH Excesses 15%	NaOH Excesses 25%
417,56	116,28	73,05	100,94

From Table 3, it can be known the value of the ion content of Mg^{2+} after the addition of NaOH reagents with variations in excesses of 0%, excesses of 15%, and excesses of 25%. When compared with the value of the content of Mg^{2+} before the addition of NaOH reagents, it was obtained that the ion content of Mg^{2+} in all excess variations decreased. But from the variation given, the best results obtained on the variation in the addition of NaOH reagent excesses 15%, this is indicated by a decrease in the ion content of Mg^{2+} from the beginning of 417.56 ppm to 73.05 ppm. This value is the smallest value when compared to the value of the ion content of Mg^{2+} in the variation in the addition of NaOH reagents excesses 0% and excesses 25%.

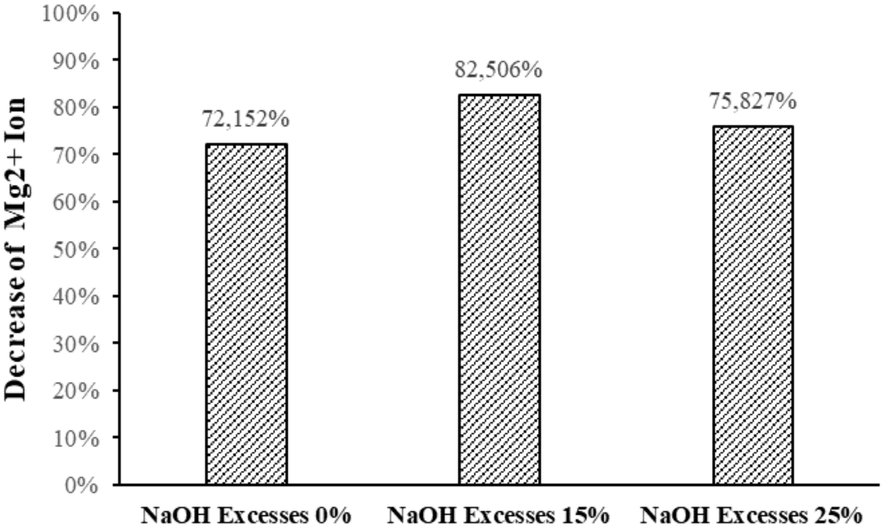

Figure 1. Decrease of Mg^{2+} Ion with Variations of NaOH Addition

Based on Fig. 1, it can be compared the percentage of the decrease in ions Mg^{2+} where NaOH excesses 0% can decrease the ion Mg^{2+} by 72.152%, for variations NaOH excesses 15% can decrease ion Mg^{2+} to 82.506% and for variations NaOH excesses 25% can reduce ion Mg^{2+} to 75.827%. According to Table 2, it has been explained that the impurities content for the ion Mg^{2+} + Ca^{2+} to match the main composition requirements of the membrane electrolysis cell by < 20 ppb, therefore a further pretreatment process is needed to reduce the content of Mg^{2+}. The solubility of magnesium salts is high in seawater [26][27]. So that the magnesium is not completely precipitated when the NaOH solution is added and the remaining Mg^{2+} ion impurities content is higher compared to the target that must be achieved.

In addition to reducing the Mg^{2+} ion, NaOH reagents also react with CO_3^{2-} ions. The effect of adding NaOH reagents on the amount of CO_3^{2-} content in RO Brine solution is presented in Table 4.

Table 4. Effect of NaOH Reagent Addition on CO_3^{2-} Ion Reduction

Content of CO_3^{2-} (ppm)			
RO Brine	NaOH Excesses 0%	NaOH Excesses 15%	NaOH Excesses 25%
0,6	0,0003	0,0000	0,0000

From Table 4, it can be known the value of the content of CO_3^{2-} ions after the addition of NaOH reagents with variations in excesses 0%, excesses 15%, and excesses 25%. When compared with the value of the content of CO_3^{2-} ions before the addition of NaOH reagents, it is obtained the result that the content of CO_3^{2-} ions decreased, even obtained the results of CO_3^{2-} ion content dropped to reach 0 ppm. From the variation given, the best results obtained on the variation in the addition of NaOH reagents excesses of 15% and 25%, this is indicated by a decrease in the content of CO_3^{2-} ions until reach 0 ppm. Meanwhile, in the variation of the addition of NaOH reagents excesses of 0% obtained results of 0.00009 ppm.

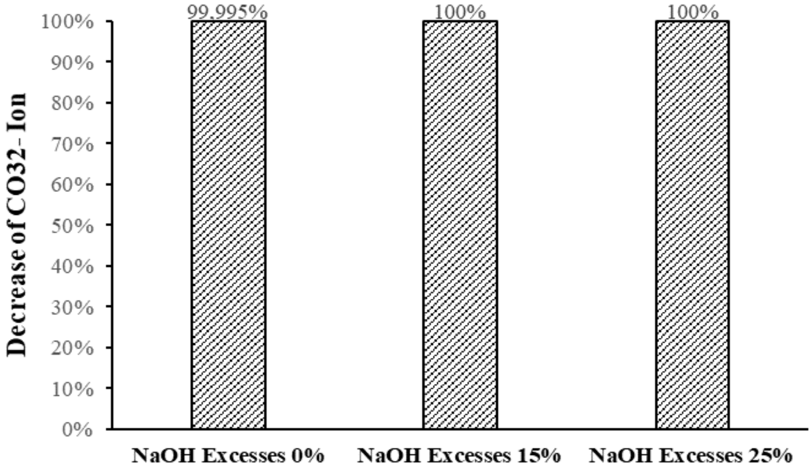

Figure 2. Decrease of CO_3^{2-} Ion with Variations of NaOH Addition

Based on Fig. 2, it can be compared the percentage of the decrease in CO_3^{2-} ions where NaOH excesses 0% can decrease CO_3^{2-} ions by 99.995%, for variations NaOH excesses 15% can decrease CO_3^{2-} ions to 100% and for variations NaOH excesses 25% can decrease CO_3^{2-} ions to 100%. Due to the relatively low initial amount of CO_3^{2-} impurity ions, the addition of reagents with excess variations did not have a significant difference. However, the NaOH reagent is used to remove 2 types of impurity ions (Mg^{2+} and CO_3^{2-}), the best NaOH reagent excess is in the 15% excess variation. This is due to the presence of sedimentation (equation 1 and 2) with the addition of NaOH excess 15%, the impurities ions can react more due to the presence of ionic force between the RO Brine particles [20].

According to Table 2, a pretreatment process is needed to reduce the content of Ca^{2+}. One of the ways to reduce the content of Ca^{2+} is by adding reagents Na_2CO_3. The addition of Na_2CO_3 to react with calcium ions Ca^{2+} to form $CaCO_3$ thus avoiding the formation of $CaSO_4$ deposits. The reactions that occur are as follows:

$Na_2CO_3 + Ca^{2+} \rightarrow CaCO_3 + 2Na^+$ (3)

This addition can be used to handle RO Brine with high alkalinity and low carbon content [20].

Table 5. Effect of Na_2CO_3 Reagent Addition on Ca^{2+} Ion Reduction

Content of Ca^{2+} [ppm]			
RO Brine	Na_2CO_3 Excesses 0%	Na_2CO_3 Excesses 20%	Na_2CO_3 Excesses 30%
116,67	19,16	17,11	14,99

From Table 5 can be known the value of the ion content of Ca^{2+} after the addition of reagents Na_2CO_3 with variations in excesses 0%, excesses 20%, and excesses 30%. When compared with the value of the ion content of Ca^{2+} before the addition, it is obtained that the ion content of Ca^{2+} decreased. From the variation given, the best results obtained on the variation in the addition of reagents Na_2CO_3 excesses 30%, this is indicated by a decrease in the ion content of Ca^{2+} to 14.99 ppm. This value is a smaller value when compared to the value of the Ca^{2+} ion content in the variation of the addition of reagent Na_2CO_3 excesses 0% and excesses 20%.

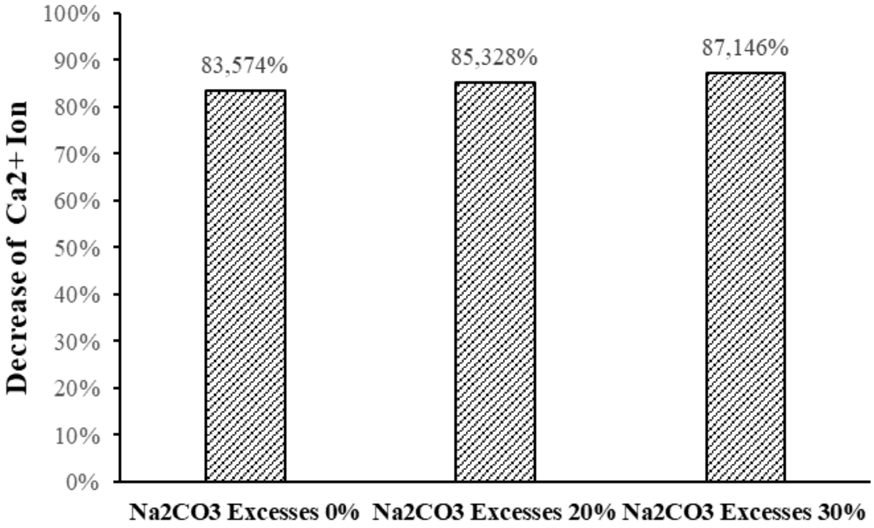

Figure 3. Decrease of Ca^{2+} Ion with Variations of Na_2CO_3 Addition

Based on Fig. 3, it can be compared the percentage of the decrease in ions Ca^{2+} where Na_2CO_3 excesses 0% can decrease the ion Ca^{2+} by 83.574%, for variations Na_2CO_3 excesses 20% can decrease the ion Ca^{2+} to 85.328% and for variations Na_2CO_3 excesses 30% can decrease the ion Ca^{2+} to 87.146%.

Then in the third stage of pretreatment process is done the addition of reagent $BaCl_2$ to reduce the content of SO_4^{2-} ions. The addition of $BaCl_2$ is done to remove the sulfate content thereby reducing calcium solids or non-carbonate solids by forming $BaSO_4$ deposits. The reaction is as follows [20]:

$SO_4^{2-} + BaCl_2 \rightarrow BaSO_{4(s)} + 2Cl^-$ (4)

$Na_2SO_4 + BaCl_2 \rightarrow BaSO_{4(s)} + 2NaCl$ (5)

Table 6 presented the results of analysis of SO_4^{2-} content in results solution of pretreatment:

Table 6. Effect of $BaCl_2$ Reagent Addition on SO_4^{2-} Ion Reduction

Experiment	SO_4^{2-} content [ppm]
1	13,8203
2	14,0484
3	13,4735
Average	13,7807

From Table 6 can be known the value of the ion content of SO_4^{2-} after the addition of $BaCl_2$ reagent with excesses, 0% decreased when compared to the initial content of SO_4^{2-} in Table 1. In the third stage pretreatment, the addition of $BaCl_2$ reagent was not using some variations of excesses because the effectiveness of $BaCl_2$ Reagent to reduce impurities has reached 96.05%.

Summary

The addition of NaOH, Na_2CO_3, and $BaCl_2$ as a reagent in the pretreatment process successfully decrease the impurities ion in RO Brine. To prevent fouling, an effective pretreatment process is needed in the solution that will be used as a feed in the electrodialysis process to remove ion impurities. The best pretreatment methods are obtained with the excess variation of NaOH reagents by 15%, Na_2CO_3 by 30% using 6% PAC coagulants, and flocculant and $BaCl_2$ excesses by 0% (without excesses). This is indicated by a decrease in the ion content of Mg^{2+} by 82.506% and CO_3^{2-} by 100% after the addition of NaOH reagents with excesses of 15%. Ion Ca^{2+} experienced a decrease of 87.146% after the addition of Na_2CO_3 reagent with 30% excesses using 6% PAC coagulant and flocculant. Then SO_4^{2-} ion decreased by 96.05% after the addition of $BaCl_2$ reagent 0% (without excesses).

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