

# Application of Response Surface Methodology to Optimise Industrial Salt Production

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Received: 23 May 2026 Accepted: 3 September 2026

**Abstract:** Common salt is mainly composed of sodium chloride (NaCl) with several impurities, such as calcium sulphate (CaSO<sub>4</sub>), magnesium sulphate (MgSO<sub>4</sub>), and magnesium chloride (MgCl<sub>2</sub>). Conventional salt purification is commonly carried out through recrystallisation. However, this method requires a double crystallisation process, and each evaporation stage may take 3–4 weeks when solar evaporation is used. Therefore, this study aims to develop a faster method for producing high-purity sodium chloride (NaCl) without double crystallisation. Chemical precipitation was selected as an alternative method to remove impurities from salt. In this process, sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) is used to remove Ca<sup>2+</sup>, sodium hydroxide (NaOH) is used to remove Mg<sup>2+</sup>, barium chloride (BaCl<sub>2</sub>) is used to remove SO<sub>4</sub><sup>2-</sup>, while anionic flocculant and sodium sulphite (Na<sub>2</sub>SO<sub>3</sub>) are used to assist sludge removal. The concentrations of these chemicals were optimised using response surface methodology (RSM). The NaCl percentage was analysed using an XRF instrument. The optimum conditions were 0.3% Na<sub>2</sub>CO<sub>3</sub>, 0.2% BaCl<sub>2</sub>, 0.061% NaOH, 5% Na<sub>2</sub>SO<sub>3</sub>, and 400 g/1000 L anionic flocculant, producing NaCl purity of 95.365%. Based on the RSM analysis, the recommended model was the cubic model, with an R<sup>2</sup> value of 0.979.

**Keywords:** Precipitation; Response Surface Methodology; Salt Purification; Salt Recovery; Sea Water

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## 1. Introduction

Indonesia still relies on imports to meet its salt demand. At the same time, national salt demand continues to increase due to population growth and industrial development [1,2]. The chlor-alkali industry, which produces sodium hydroxide (NaOH) and chlorine gas (Cl<sub>2</sub>), requires high-purity sodium chloride (NaCl) as a raw material [2,3]. However, NaCl for the chlor-alkali industry in Indonesia is still largely imported from Australia and other countries. The food industry also requires salt with suitable purity and low impurity content [4].

Salt can be produced from saline water using four main technologies. The first is thermal processing, such as solar distillation, multi-effect distillation, multiple-effect evaporation, and

multi-stage flash distillation. The second is membrane processing, including reverse osmosis and electrodialysis or electrodialysis reversal. The third is chemical processing, such as ion exchange, reactive precipitation, and calcination. The fourth is hybrid processing, which combines two or more methods, such as membrane distillation and membrane crystallisation [1–3], [5–10].

The chlor-alkali industry requires high-purity salt with low concentrations of impurities such as  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ . These impurities can interfere with  $\text{Na}^+$  ion transport across the membrane in the electrolysis reactor. Conventional salt purification is commonly carried out through recrystallisation combined with chemical precipitation. Astuti et al. [11] applied recrystallisation and chemical precipitation to raw salt using  $\text{Na}_2\text{CO}_3$  as a precipitating agent. Their results showed that  $\text{Na}_2\text{CO}_3$  reduced Ca content from 0.03% to 0.01% at stirring times of 60–150 min. Tarmidzi et al. [2] produced food-grade salt using chemical precipitation with  $\text{CaCO}_3$  and  $\text{Li}_2\text{CO}_3$  to remove Mg and Ca impurities.

However, recrystallisation commonly requires a double crystallisation process, and each evaporation stage may take 3–4 weeks when solar evaporation is used. This makes the process slow and less efficient. Chemical precipitation offers a faster alternative because it can remove impurities through selective reactions without requiring repeated crystallisation. Therefore, this method has potential for producing purified salt suitable for chlor-alkali applications.

In this study, industrial salt was produced from seawater using a five-step chemical precipitation process. The chemicals were used to bind and precipitate impurities, removing the need for recrystallisation. The target impurities included calcium sulphate ( $\text{CaSO}_4$ ), magnesium sulphate ( $\text{MgSO}_4$ ), magnesium chloride ( $\text{MgCl}_2$ ), and sludge. These impurities were removed using sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), barium chloride ( $\text{BaCl}_2$ ), sodium sulphite ( $\text{Na}_2\text{SO}_3$ ), sodium hydroxide ( $\text{NaOH}$ ), and anionic flocculant. Response surface methodology was used to optimise the composition of these chemicals [12–14].

## 2. Materials and Methods

### 2.1 Materials and tools

The main ingredients in this research are sea water found on the white sand beach of South Lampung, sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), technical Barium Chloride ( $\text{BaCl}_2$ ), and technical sodium sulfite ( $\text{Na}_2\text{SO}_3$ ), technical sodium hydroxide ( $\text{NaOH}$ ), technical anionic flocculant. The tools used in this research are Beaker glass, filter paper, stirring rod, digital scale, stove, stainless steel container, and XRF Epsilon 3 XLE instrument.

### 2.2 Method

The research started with optimisation of the process of making sodium chloride from seawater with the first stage making the formulation and response design, followed by the formulation stage, then response analysis, and the last stage was optimization. The experimental procedure was further defined based on the response surface methodology design. The fixed experimental conditions were the seawater volume and processing time, while the independent variables were the concentrations of  $\text{Na}_2\text{CO}_3$ ,  $\text{BaCl}_2$ ,  $\text{Na}_2\text{SO}_3$ ,  $\text{NaOH}$ , and anionic flocculant. These variables were selected because they directly influence the precipitation of calcium, magnesium, sulphate, and suspended impurities during salt purification. The experimental design was generated using Design Expert 7.0, and 32 treatment combinations were obtained from the RSM design. The percentage of NaCl in the final salt product was used as the main response variable.

### *2.3 Stage of making formulation and response design*

Formulation and response design was made using the DX 7.0® Program to determine the fixed and independent variables. In this study, the fixed variables were the time and volume of seawater used. Meanwhile, the independent variable is the concentration of the addition of chemicals, namely,  $\text{Na}_2\text{CO}_3$ ,  $\text{BaCl}_2$ ,  $\text{Na}_2\text{SO}_3$ ,  $\text{NaOH}$ , and flocculants.

Minimum and maximum threshold values are entered into the DX 7® RSM Central Composite Design program for randomisation. After randomisation of combinations, 32 treatments were obtained to be analysed (Table 1) and the measured and optimised response was %NaCl. The purity of NaCl was analysed using an X-ray fluorescence (XRF) instrument (XRF InnovX Olympus, USA). The optimum salt product and industrial salt (Made in Australia) were analysed using XRF and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) from Plasmaquant PQ 9100 elite (USA).

Each experimental run was prepared using 500 mL of seawater. The chemical dosages were added according to the treatment combination generated by the RSM design. The purification process involved sequential chemical precipitation using  $\text{BaCl}_2$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaOH}$ , anionic flocculant, and  $\text{Na}_2\text{SO}_3$ , followed by decantation, filtration, and evaporation to obtain the salt product. This sequence was applied consistently for all experimental runs to ensure that the effect of the chemical concentration variables could be evaluated systematically.

### *2.4 Formulation stage*

The formulation stage was the stage of making sodium chloride according to the process conditions (formula) in Table 1. Each formula was made of NaCl from 500 ml of seawater. The manufacturing process was carried out by the process presented in Figure 1. Next, the sample characterisation using XRF was carried out in stages [12]. The sample was in a powder state and sieved with a 200 sieve; the sample was tested using XRF, the XRF tool cover was opened, and each sample was placed in the sample holder. The analysis using XRF was to get a response, namely the level of NaCl in the product.

The NaCl purity of each salt product was determined using X-ray fluorescence spectroscopy. Before analysis, the dried salt product was prepared in powder form and sieved using a 200-mesh sieve to improve sample uniformity. The prepared sample was then placed in the XRF sample holder and analysed to determine the elemental composition and NaCl purity. For comparison and confirmation, the optimum salt product and the reference industrial salt were also analysed using XRF and inductively coupled plasma optical emission spectroscopy.

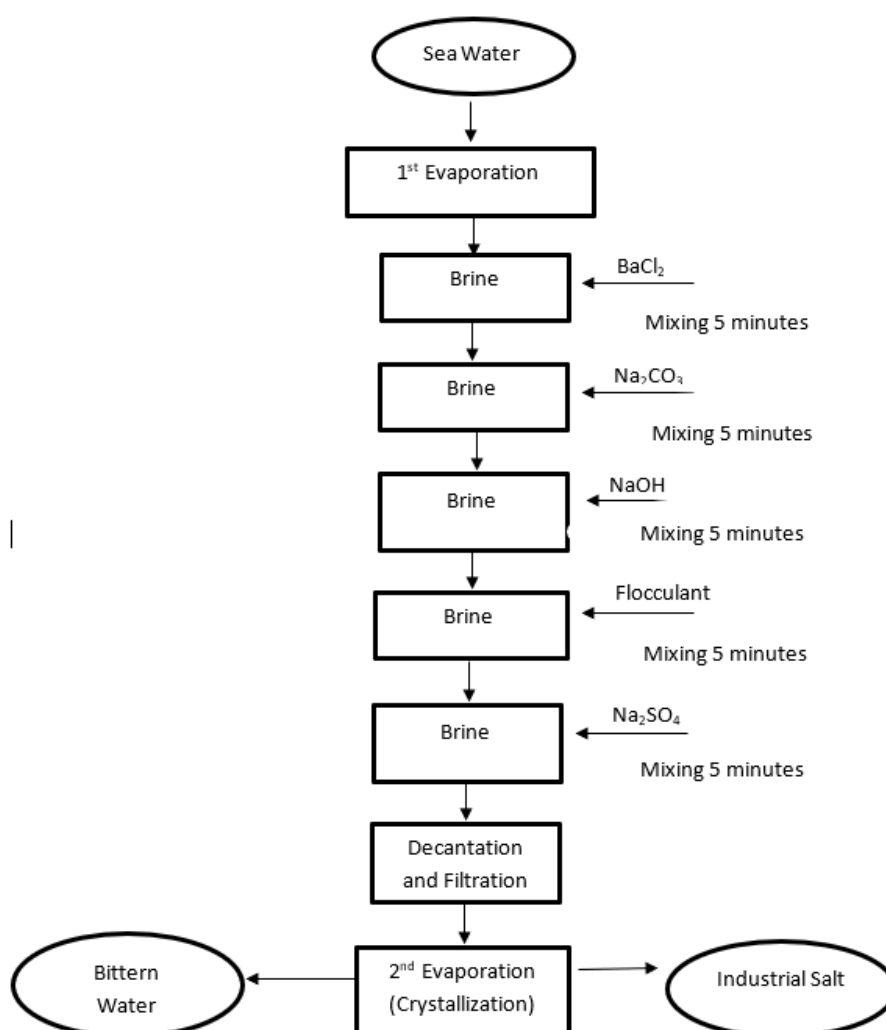
### *2.5 Response analysis stage*

Each response variable was then analysed by ANOVA. The ANOVA model used can be chosen as suggested by the program, namely the model that has the highest level and produces a significant ANOVA value. The ANOVA models contained in this design are Linear, 2FI, Quadratic, and Cubic. The model that gave significance to the ANOVA and non-significance to the lack of fit was chosen to analyse the variables. In addition, the DX 7.0® program also provides a normal plot of the residual facility (normal plot of residual) which indicates whether the residual (difference between the actual response and the predicted response value) follows the normal line (straight line). Data points that are closer to the normality line indicate data that is normally distributed, which means the actual results will be close to the results predicted by the DX 7.0® program.

Model validation was carried out using several statistical indicators from the RSM analysis. The selected model was evaluated based on ANOVA significance, p-value, coefficient of determination, lack-of-fit test, and residual normality. A model was considered acceptable when the ANOVA result was significant, the lack of fit was not significant, and the residuals followed an approximately normal distribution. The desirability function was then used during the optimisation stage to identify the chemical-dosage combination that maximised NaCl purity within the tested experimental range.

## 2.6 Optimisation stage

In the Process Optimization stage, each response (%NaCl) was determined for its optimisation objective in the DX 7.0® program. This program would perform optimisation according to the input variable data and response measurement data. The output of the optimisation stage is the recommendation of several new formulas that are optimal according to the program. The most optimal formula is the formula with the maximum desirability value. The desirability value is the value of the optimisation objective function which shows the program's ability to fulfill the wishes based on the criteria set in the final product. The value ranges from 0 to 1.0. The desirability value, which is getting closer to 1.0, indicates the program's ability to produce the desired product is getting more perfect. The purpose of optimisation is not to obtain a desirability value of 1.0, but to find the best conditions that meet all objective functions [14].



**Figure 1.** Diagram flow on RSM

### 3. Results and discussion

#### 3.1. NaCl level response analysis

From Table 1, it can be seen that the 6th experiment shows the highest %NaCl (Sodium Chloride) which is 90.6% with the addition of  $\text{Na}_2\text{CO}_3$  (Sodium Carbonate) 0.35%,  $\text{BaCl}_2$  (Barium Chloride) 0.05%,  $\text{Na}_2\text{SO}_3$  (Sodium Sulphide) 2.5 %, NaOH (Sodium Hydroxide) 0.02 %, and flocculant 100 g/L. While the lowest % NaCl levels were found in the 13th experiment, namely 78.505% with the addition of 0.2%  $\text{Na}_2\text{CO}_3$  (Sodium Carbonate), 0.1%  $\text{BaCl}_2$  (Barium Chloride),  $\text{Na}_2\text{SO}_3$  (Sodium Sulphide) 10%, NaOH (Sodium Hydroxide) 0, 02 %, and 600 g/L flocculant. The results in the table were then analysed using Software Design Expert 7.0.0 (Trial Version) to determine the optimum conditions and model equations.

**Table 1.** RSM Experimental Design and Response Results

| Run | $\text{Na}_2\text{CO}_3$ | $\text{BaCl}_2$ | $\text{Na}_2\text{SO}_3$ | NaOH | Flokulan | %NaCl  |
|-----|--------------------------|-----------------|--------------------------|------|----------|--------|
| 1   | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 87.972 |
| 2   | 0.20                     | 0.10            | 5.00                     | 0.06 | 600      | 87.085 |
| 3   | 0.20                     | 0.20            | 10.00                    | 0.06 | 600      | 83.438 |
| 4   | 0.10                     | 0.10            | 10.00                    | 0.06 | 600      | 86.880 |
| 5   | 0.10                     | 0.20            | 5.00                     | 0.02 | 400      | 88.791 |
| 6   | 0.35                     | 0.05            | 2.50                     | 0.02 | 100      | 90.620 |
| 7   | 0.05                     | 0.05            | 12.50                    | 0.02 | 100      | 88.508 |
| 8   | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 88.889 |
| 9   | 0.10                     | 0.10            | 10.00                    | 0.02 | 400      | 84.778 |
| 10  | 0.05                     | 0.05            | 2.50                     | 0.02 | 900      | 88.058 |
| 11  | 0.25                     | 0.05            | 2.50                     | 0.02 | 100      | 89.698 |
| 12  | 0.10                     | 0.10            | 5.00                     | 0.02 | 600      | 89.556 |
| 13  | 0.20                     | 0.10            | 10.00                    | 0.02 | 600      | 78.505 |
| 14  | 0.10                     | 0.20            | 10.00                    | 0.02 | 600      | 82.222 |
| 15  | 0.05                     | 0.05            | 2.50                     | 0.06 | 100      | 88.070 |
| 16  | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 87.124 |
| 17  | 0.10                     | 0.20            | 5.00                     | 0.06 | 600      | 89.290 |
| 18  | 0.05                     | 0.05            | 2.50                     | 0.02 | 1100     | 88.849 |
| 19  | 0.20                     | 0.20            | 5.00                     | 0.06 | 400      | 90.365 |
| 20  | 0.20                     | 0.10            | 10.00                    | 0.06 | 400      | 87.232 |
| 21  | 0.05                     | 0.35            | 2.50                     | 0.02 | 100      | 89.046 |
| 22  | 0.10                     | 0.20            | 10.00                    | 0.06 | 400      | 88.979 |
| 23  | 0.20                     | 0.20            | 5.00                     | 0.02 | 600      | 87.428 |
| 24  | 0.05                     | 0.05            | 17.50                    | 0.02 | 100      | 84.046 |
| 25  | 0.20                     | 0.10            | 5.00                     | 0.02 | 400      | 88.076 |
| 26  | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 89.430 |
| 27  | 0.20                     | 0.20            | 10.00                    | 0.02 | 400      | 86.782 |
| 28  | 0.05                     | 0.05            | 2.50                     | 0.10 | 100      | 88.229 |
| 29  | 0.10                     | 0.10            | 5.00                     | 0.06 | 400      | 86.505 |
| 30  | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 89.430 |
| 31  | 0.05                     | 0.05            | 2.50                     | 0.02 | 100      | 87.942 |
| 32  | 0.05                     | 0.25            | 2.50                     | 0.02 | 100      | 88.377 |

The 32 experimental runs in Table 1 represent the complete RSM experimental matrix used to evaluate the influence of the five chemical additives on NaCl purity. The response values were obtained from XRF analysis and were used directly for model fitting in Design Expert 7.0. The experimental matrix included different combinations of  $\text{Na}_2\text{CO}_3$ ,  $\text{BaCl}_2$ ,  $\text{Na}_2\text{SO}_3$ , NaOH, and flocculant concentrations, allowing the individual and interaction effects of these chemicals on NaCl purity to be assessed through the RSM model.

The polynomial model recommended by Design Expert 7.0. program is cubic by showing the R-squared value that is close to 1, namely 0.9780. The cubic model of this process is shown in Equation 1. Where A is  $\text{Na}_2\text{CO}_3$  (%), B is  $\text{BaCl}_2$  (%), C is  $\text{Na}_2\text{SO}_3$  (%), D is NaOH (%) and E is flocculant (g/1000 lt). Compared to other models, the cubic model produces an R-Squared value highest. In addition, this model is significant with a p-value < F smaller than 0.05 (0.0271) on responses. Analyses of the variant are in Table 2. The lack of fit of this analysis is not significant as listed in Table 2. The non-significant lack of fit is good since the mathematical

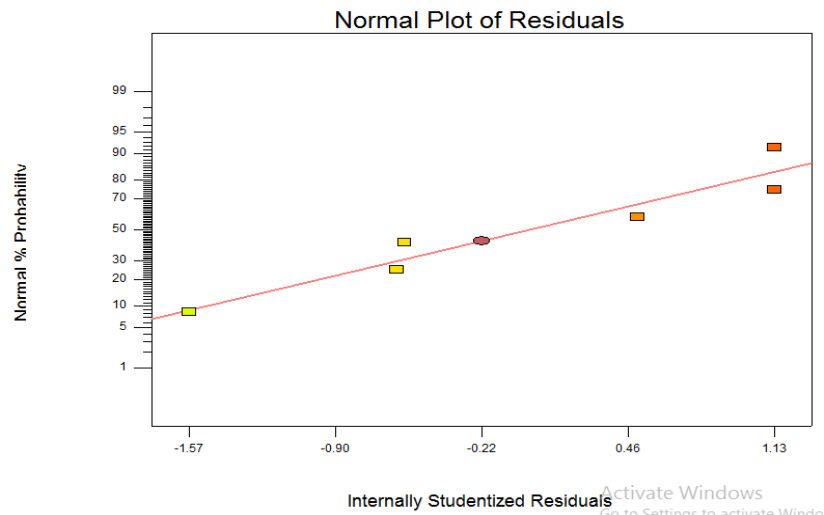
model is useful for fixing the optimum value. This model is useful to predict salt purity using concentrate chemicals in the precipitation process.

$$\%NaCl = 87.51 + 0.052*A + 0.005042*B - 0.77*C + 0.92D + 0.0069*E - 0.91*AB + 0.93*AC - 0.16*AD - 0.89*AE + 0.24*BC + 0.13*BD = 0.97*BE + 0.70*CD + 1.65*CE - 0.059*DE \quad (1)$$

The normal graph plot of residuals in Figure 2 indicates the relationship between the actual value and the predicted value, the picture shows the normality plot of the residuals following the normal line (straight line) which explains that the data points that are getting closer to the normality line show data that is normally distributed, which means the results actual results will approach the results predicted by the DX 7.0® program [12].

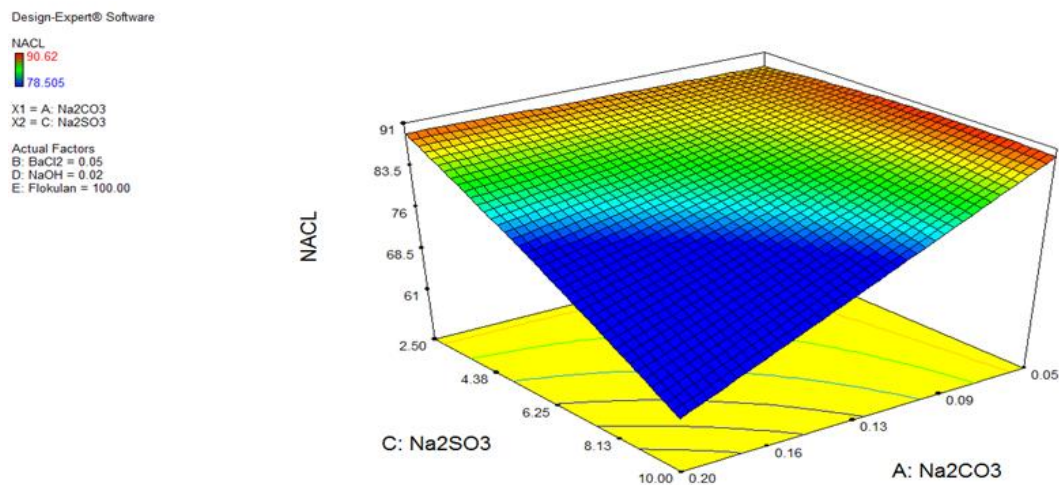
**Table 2.** Analysis of variant

| Source                                | Sum of Squares | df | Mean Square           | F Value               | p-value Prob > F |                 |
|---------------------------------------|----------------|----|-----------------------|-----------------------|------------------|-----------------|
| <b>Model</b>                          | 142.62         | 15 | 9.51                  | 2.73                  | 0.0271           | significant     |
| <b>A-Na<sub>2</sub>CO<sub>3</sub></b> | 0.066          | 1  | 0.066                 | 0.019                 | 0.8921           |                 |
| <b>B-BaCl<sub>2</sub></b>             | 6.100E-004     | 1  | 6.1x 10 <sup>-4</sup> | 1.75x10 <sup>-4</sup> | 0.9896           |                 |
| <b>C-Na<sub>2</sub>SO<sub>3</sub></b> | 14.17          | 1  | 14.17                 | 4.08                  | 0.0606           |                 |
| <b>D-NaOH</b>                         | 20.50          | 1  | 20.50                 | 5.90                  | 0.0273           |                 |
| <b>E-Flocculant</b>                   | 0.11           | 1  | 0.11                  | 0.033                 | 0.8590           |                 |
| <b>AB</b>                             | 13.13          | 1  | 13.13                 | 3.78                  | 0.0698           |                 |
| <b>AC</b>                             | 13.75          | 1  | 13.75                 | 3.95                  | 0.0641           |                 |
| <b>AD</b>                             | 0.41           | 1  | 0.41                  | 0.12                  | 0.7358           |                 |
| <b>AE</b>                             | 12.79          | 1  | 12.79                 | 3.68                  | 0.0732           |                 |
| <b>BC</b>                             | 0.91           | 1  | 0.91                  | 0.26                  | 0.6161           |                 |
| <b>BD</b>                             | 0.26           | 1  | 0.26                  | 0.074                 | 0.7887           |                 |
| <b>BE</b>                             | 15.09          | 1  | 15.09                 | 4.34                  | 0.0536           |                 |
| <b>CD</b>                             | 7.76           | 1  | 7.76                  | 2.23                  | 0.1545           |                 |
| <b>CE</b>                             | 43.62          | 1  | 43.62                 | 12.55                 | 0.0027           |                 |
| <b>DE</b>                             | 0.055          | 1  | 0.055                 | 0.016                 | 0.9012           |                 |
| <b>Residual</b>                       | 55.62          | 16 | 3.48                  |                       |                  |                 |
| <b>Lack of Fit</b>                    | 34.34          | 11 | 3.12                  | 0.73                  | 0.6904           | not significant |
| <b>Pure Error</b>                     | 21.28          | 5  | 4.26                  |                       |                  |                 |
| <b>Cor Total</b>                      | 198.24         | 31 |                       |                       |                  |                 |



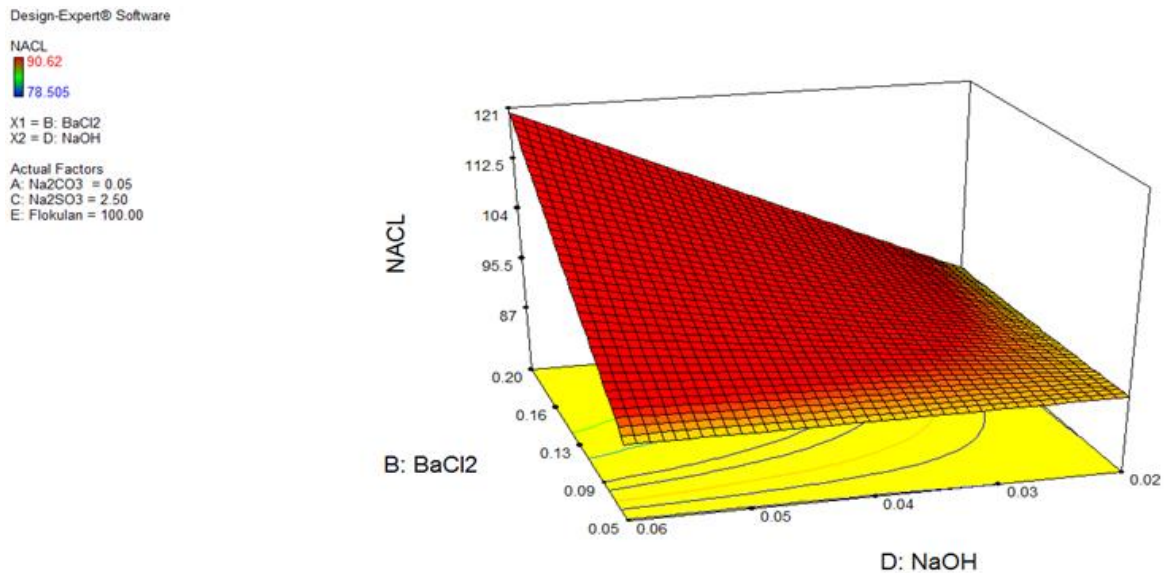
**Figure 2.** The normal plot of residuals

In general, the response surface can be visualized in the form of graphics or contour plots. This graph is very helpful to see the shape of the response surface, which is in the form of a maximum point of response, and a minimum point of response. The graph shows how the combination of components affects the response of % NaCl. Different colors on the counterplot graph indicate the response value of % NaCl. Figure 3 shows the relationship between variables A and C with the actual vector variables B, D, and E. The counter-plot and 3D Surface graphs show the response values. The blue color indicates the lowest response value, which is 65.8535, with a darker color change which indicates a higher value of the resulting response.



**Figure 3.** 3D surface AC variable on responses

Figure 4 shows the relationship between variables B and D variables with the actual variables A, C, and E. The counter-plot and 3D Surface graphs show the value of the response. The orange color shows the lowest response value, namely at 90.0606 the color change is getting darker which indicates the higher the response value is generated [14].



**Figure 4.** 3D surface variable BD on responses

### 3.2. Process optimisation

In the Process Optimization stage, each response (%NaCl) is determined for its optimisation objective in the DX 7.0® program. This program will perform optimisation according to the input variable data and response measurement data. The output of the optimisation stage is the recommendation of several new formulas that are optimal according to the program. The most optimal formula is the formula with the maximum desirability value.

The desirability value is the value of the optimisation objective function which shows the program's ability to fulfill the wishes based on the criteria set in the final product. The value ranges from 0 to 1.0. The desirability value, which is getting closer to 1.0, indicates the program's ability to produce the desired product is getting more perfect. The purpose of optimisation is not to obtain a desirability value of 1.0, but to find the best conditions that bring together all objective functions [13].

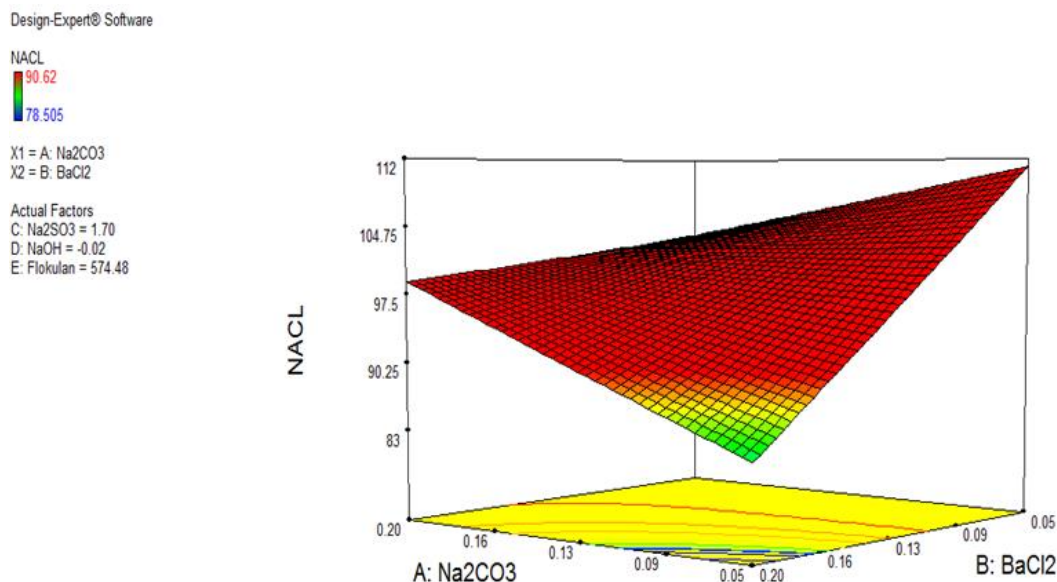
The addition of Na<sub>2</sub>CO<sub>3</sub>, BaCl<sub>2</sub>, Na<sub>2</sub>SO<sub>3</sub>, NaOH, and flocculants was optimised with a predetermined range, with an importance level of 3(+++). The addition of these materials affects the %NaCl obtained. The % NaCl response is an optimised response with a maximum target of 95% with an importance level of 5(+++). % NaCl 95% will match the NaCl needed in the industrial world. Based on the optimisation process, the DX 7.0® program provides 23 optimal formula solutions which can be seen in Table 3.



**Table 3.** Optimised component

| No. | Na <sub>2</sub> CO <sub>3</sub> | BaCl <sub>2</sub> | Na <sub>2</sub> SO <sub>3</sub> | NaOH  | Flocculant | NaCl   | Desirability |
|-----|---------------------------------|-------------------|---------------------------------|-------|------------|--------|--------------|
| 1   | 0.050                           | 0.040             | 3.930                           | 0.010 | 188.950    | 94.998 | 1            |
| 2   | 0.030                           | 0.080             | 3.750                           | 0.020 | 375.900    | 95.002 | 1            |
| 3   | 0.040                           | 0.020             | 0.240                           | 0.010 | 239.940    | 95.000 | 1            |
| 4   | 0.160                           | 0.130             | 0.890                           | 0.020 | 449.360    | 95.000 | 1            |
| 5   | 0.160                           | 0.060             | 3.120                           | 0.000 | 448.890    | 94.998 | 1            |
| 6   | 0.080                           | 0.190             | 3.890                           | 0.040 | 352.080    | 95.001 | 1            |
| 7   | 0.180                           | 0.110             | 3.980                           | 0.010 | 38.440     | 95.000 | 1            |
| 8   | 0.010                           | 0.000             | 4.970                           | 0.010 | 211.100    | 94.999 | 1            |
| 9   | 0.060                           | 0.020             | 2.800                           | 0.050 | 323.660    | 94.999 | 1            |
| 10  | 0.140                           | 0.170             | 1.700                           | 0.020 | 574.480    | 95.002 | 1            |
| 11  | 0.080                           | 0.140             | 6.740                           | 0.020 | 592.960    | 94.999 | 1            |
| 12  | 0.000                           | 0.020             | 2.100                           | 0.010 | 336.710    | 95.001 | 1            |
| 13  | 0.000                           | 0.070             | 2.640                           | 0.010 | 527.890    | 95.002 | 1            |
| 14  | 0.030                           | 0.120             | 0.080                           | 0.040 | 156.700    | 95.001 | 1            |
| 15  | 0.060                           | 0.150             | 4.690                           | 0.040 | 308.820    | 94.999 | 1            |
| 16  | 0.030                           | 0.000             | 4.280                           | 0.010 | 56.890     | 95.365 | 1            |
| 17  | 0.120                           | 0.160             | 5.760                           | 0.020 | 273.650    | 95.001 | 1            |
| 18  | 0.030                           | 0.180             | 1.730                           | 0.040 | 366.190    | 94.999 | 1            |
| 19  | 0.180                           | 0.040             | 0.620                           | 0.020 | 160.700    | 94.999 | 1            |
| 20  | 0.030                           | 0.070             | 7.080                           | 0.040 | 108.250    | 95.002 | 1            |
| 21  | 0.050                           | 0.120             | 1.050                           | 0.060 | 198.320    | 94.999 | 1            |
| 22  | 0.020                           | 0.070             | 3.640                           | 0.020 | 512.670    | 95.000 | 1            |
| 23  | 0.120                           | 0.020             | 2.190                           | 0.030 | 355.160    | 94.999 | 1            |

It was found that the desirability value showed at number 1 and from the table, it was also found which solution could obtain the highest %NaCl. In this research, solution number 16 was chosen with a prediction of NaCl 95.365 % with 3D surface NaCl Prediction in Figure 5.



**Figure 5.** 3D surface of NaCl prediction

### 3.3. Decrease in impurity levels in NaCl

The purpose of this study was to obtain the results of % NaCl with a high level of purity by reducing the levels of impurities in Sodium Chloride (NaCl). Impurities in salt or NaCl are not desirable because they will interfere with the process in subsequent production. The presence of high levels of impurities also reduces the purity of sodium chloride (salt). The decrease of impurities was listed in Table 4.

**Table 4.** Solution formula on RSM

| RUN | Decreasing of Mg | Decreasing of Ca | Decreasing of S |
|-----|------------------|------------------|-----------------|
| 1   | -10.5187         | 17.8079          | -11.5675        |
| 2   | 11.2391          | 23.9664          | -9.8910         |
| 3   | -35.4466         | 17.3341          | -96.4376        |
| 4   | -20.0288         | 44.8751          | -70.2850        |
| 5   | 16.9308          | 27.6701          | -10.7712        |
| 6   | 52.8818          | 29.3066          | 28.3319         |
| 7   | -15.0576         | 70.0473          | -66.1777        |
| 8   | 3.45821          | 29.8449          | 7.2087          |
| 9   | -39.26512        | 47.6313          | -93.4619        |
| 10  | 36.3112          | 7.40740          | 1.1735          |
| 11  | 22.1181          | 34.1515          | 12.4057         |
| 12  | -7.3487          | 65.7838          | -11.0226        |
| 13  | -87.8962         | 15.8484          | -141.5340       |
| 14  | -22.7665         | 27.0241          | -94.2582        |
| 15  | -12.0317         | 21.8346          | 0.2933          |
| 16  | -3.6023          | 15.5038          | -10.1844        |
| 17  | 23.9913          | 32.2566          | 7.5020          |
| 18  | 14.1210          | 8.91472          | 4.4006          |
| 19  | 18.0835          | 51.0335          | 1.2573          |
| 20  | -15.9221         | 55.9000          | -86.5046        |
| 21  | 14.1930          | 31.2661          | 5.1550          |
| 22  | 71.3976          | 40.7622          | -23.0092        |
| 23  | 6.6282           | 20.8871          | -15.0461        |
| 24  | -0.7204          | 16.2575          | -105.4480       |
| 25  | 14.5533          | 24.2463          | -4.3587         |
| 26  | 33.2132          | 26.9810          | 13.9145         |
| 27  | -14.1930         | 35.8527          | -38.7678        |
| 28  | 11.6714          | 34.4962          | 8.1726          |
| 29  | 18.8760          | 11.5202          | -20.5365        |
| 30  | 33.2132          | 26.9810          | 13.9145         |
| 31  | 21.5417          | 36.7140          | 1.0058          |
| 32  | 7.7809           | 26.4211          | -17.5189        |

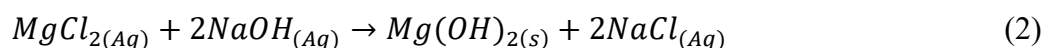
The impurity removal results show that the purification performance was controlled by the combined effect of the chemical additives rather than by one chemical alone. NaOH contributed mainly to Mg removal through the formation of  $Mg(OH)_2$  precipitate, while  $Na_2CO_3$  promoted Ca removal through  $CaCO_3$  formation.  $BaCl_2$  supported sulphate removal through  $BaSO_4$  precipitation. However, the highest removal of Mg, Ca, and sulphate did not occur under the same experimental run because each additive affected the precipitation equilibrium differently. This indicates that the optimum salt purification condition should be selected based on overall NaCl purity and desirability, rather than the maximum removal of a single impurity.

Some experimental runs produced negative impurity removal values, which indicate that the measured impurity level after treatment was higher than the reference value. This may be caused by incomplete precipitation, interaction between chemical additives, sample matrix effects during analysis, or experimental variability. These values were retained because they represent the actual measured response under the tested RSM conditions. Therefore, the impurity removal trends should be interpreted together with the NaCl purity response, not as isolated removal indicators.

### 3.4. Decrease in Mg levels in NaCl

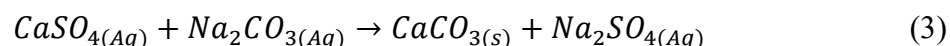
Referring to the chemical reaction in the framework of thought, in reducing the level of atomic impurities, Mg seawater is reacted with sodium hydroxide (NaOH) to obtain  $Mg(OH)_2$  in solid which will then be precipitated by the flocculant. The graph of the decrease in Mg levels can be seen in Table 4. The table shows the results of the highest decrease in Mg levels, namely in

the 22<sup>nd</sup> experiment with a decrease of 71.39%. The NaOH is to bind Mg as shown in following equation:



### 3.5. Decrease in Ca levels in NaCl

Referring to the chemical reaction in the framework of thought, in decreasing the level of Ca impurities, seawater is reacted with Sodium Carbonate (Na<sub>2</sub>CO<sub>3</sub>) to get CaCO<sub>3</sub> in a solid which will then be precipitated by the flocculant [2,3,11]. The graph of the decrease in Ca levels is shown in Table 4. The table shows the results of the highest decrease in Ca levels, namely in the 7<sup>th</sup> experiment with a decrease of 70.047%. Na<sub>2</sub>CO<sub>3</sub> to bind Ca.



### 3.6. Decreases in S levels

Referring to the chemical reaction in the framework of thought, in decreasing the level of SO<sub>4</sub> impurities, seawater is reacted with Barium chloride (BaCl<sub>2</sub>) to get BaSO<sub>4</sub> in a solid which will then be precipitated by the flocculant [2]. The graph of the decrease in S levels can be seen in Table 4. The table shows the results of the highest decrease in S levels, namely in the 6<sup>th</sup> experiment, with a decrease of 28.3319%. BaCl<sub>2</sub> binds SO<sub>4</sub>.



The findings suggest that chemical precipitation can improve salt purity when the dosage combination is properly controlled. The RSM result shows that the optimum formulation was not necessarily the condition with the highest individual impurity removal, but the condition that produced the best balance between impurity reduction and NaCl purity. This highlights the importance of using optimisation tools such as RSM to evaluate multi-chemical systems where several reactions occur simultaneously.

Direct numerical comparison with previous studies was not conducted because it is outside the scope of the present work. The objective of this study was to optimise the chemical dosage combination for the current seawater sample using RSM, rather than to benchmark different salt purification methods. Since seawater composition, impurity levels, treatment sequence, chemical dosage range and analytical methods vary between studies, direct comparison of impurity removal percentages may be misleading. Therefore, the discussion focuses on interpreting the impurity removal trends observed in the present experimental dataset.

## 4. Conclusions

The model of NaCl purity is a cubic model as shown in equation 1. The methodology used a 32-run response surface methodology design with Na<sub>2</sub>CO<sub>3</sub>, BaCl<sub>2</sub>, Na<sub>2</sub>SO<sub>3</sub>, NaOH, and anionic flocculant as independent variables, while seawater volume and processing time were kept constant. NaCl purity was measured using XRF, and the optimum product was further compared with industrial salt using XRF and ICP-OES. The RSM model was validated using ANOVA, coefficient of determination, lack-of-fit analysis, residual normality, and desirability evaluation. These methodological details support the reproducibility of the salt-purification and optimisation procedure. The model showed good predictive performance for NaCl purity based on the chemical dosages used in the precipitation process, with an R<sup>2</sup> value of 0.9789. Based on the RSM analysis, the optimum condition for achieving the highest NaCl purity was 0.03% Na<sub>2</sub>CO<sub>3</sub>, 0% BaCl<sub>2</sub>, 4.28% NaOH, 0.01% Na<sub>2</sub>SO<sub>3</sub>, and 56.890 g/1000 L flocculant. This condition produced a predicted NaCl purity of 95.365%, as shown in Table 3. The reference

industrial salt from Australia had 99% purity based on ICP-OES analysis. However, when analysed using XRF, the same industrial salt showed 95.4% NaCl purity. Therefore, the salt product produced in this study showed comparable XRF-based NaCl purity to the reference industrial salt from Australia. From the research that has been done, there are suggestions for further research, namely by using seawater samples with good conditions, there is no pollution level around the sampling so that purer NaCl results are obtained.

## Acknowledgement

The authors gratefully acknowledge the support from the Research Centre for Mining Technology, National Research and Innovation Agency (BRIN), Politeknik Negeri Lampung, Universiti Sains Malaysia, and Universiti Teknologi MARA. The contributions and facilities provided by these institutions were essential in completing this research. We appreciate the collaboration and support that have enriched this study.

## Conflict of interest

The authors declare no conflicts of interest in publishing this paper. Authors have the option to disclose any potential conflicts of interest.

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