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Development of Efficient and Scalable Production Process of Analytical Grade Sodium Chloride at Laboratory Scale

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Abstract. Analytical grade Sodium chloride (NaCl) is one type of salt with a purity of > 99.50 % NaCl content, including other impurities. In laboratories, salt is used as a reagent and a standard solution. This research carried out the salt production process through purification, acidification, neutralization, crystallization, and drying methods. Compared to existing technologies, the advantages of this technology are its simplicity and ability to produce accompanying products. In the first stage of the purification process, the precipitation of sulfate (SO_4^{2-}) and magnesium (Mg^{2+}) ions was examined. In this step, 20 % w/v barium chloride ($BaCl_2$) and 20 % w/v sodium hydroxide (NaOH) solution were consecutively used, and after 12 hours of sitting, the filtering process separated the mixture. The second stage of purification will investigate the precipitation of calcium (Ca^{2+}) ions using 20 % w/v sodium carbonate (Na_2CO_3) solution, and the mixture was again separated by filtering after 12 hours. All stages of the purification process have shown that the ions concentration decreases by adding $BaCl_2$, NaOH, and Na_2CO_3 solution. Furthermore, the acidification, neutralization, and crystallization processes can control pH and potassium (K^+) content. The results demonstrated that the technology produced 30% analytical grade NaCl with purity > 99.50% and high brine solution as a by-product, which can be further processed for producing pharmaceutical and food-grade salts.

Keywords: Analytical grade; Development; Production process; Purification; Sodium chloride

1. Introduction

Sodium Chloride (NaCl) or salt is an essential commodity. It can be used as a raw material or a mixture in various fields, including chemical, agricultural, animal husbandry, pharmaceutical, health, cosmetic, food, and beverage industries. In the chemical industry, salt is the primary raw material for the production of chlorine gas (Cl_2), hydrochloric acid (HCl), sodium hydroxide (NaOH), sodium sulfate (Na_2SO_4), sodium carbonate (Na_2CO_3), and sodium bicarbonate ($NaHCO_3$) (Ono, and Erhard, 2012; Sedivy, 2010). Salt is widely used in leather tanning, fish preservation, and oil drilling. Moreover, it is also used in weather modification through seeding and cloud formation to form artificial rain. Types of salt suitable for use include analytical, pharmaceutical, industrial, food, and miscellaneous grade salts. Food-grade salt is known for making salted fish-tanning skin (Ono, and Erhard, 2012; Sedivy, 2009).

Analytical grade NaCl is a high-purity salt with a NaCl content of 99.50 - 100.50% and

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a low content of other compounds. The most common use of salt is in laboratory chemicals. Its common uses include chemical reagents, standard sodium ion in absorption atomic spectrophotometers, standardization of $AgNO_3$ solutions in argentometry volumetric, microbiological media, material synthesis, cell microbiology, and ion chromatography.

NaCl is also used for special purposes in the synthesis of material. The effect of hydrothermal reaction time and NaCl addition on nano rosette TiO_2 crystal growth characteristics during hydrothermal reaction has been examined. With no addition of NaCl, the nano rosette forms at full growth, indicated by the high intensity of the crystal structure indexed to rutile P42/mnm with lattice parameters of a = 4.557(6) Å and c = 2.940(5) Å. The cross-sectional rosette petal grew to 250 nm. On the contrary, the crystal growth during hydrothermal reaction could be controlled with the addition of NaCl. In this work, with the addition of 2.5% v/v NaCl, the cross-sectional rosette size was only about 50 nm, 80% smaller than that of the crystal grown with no NaCl addition (Sofyan *et al.,* 2019).

Analytical grade NaCl specifications refer to Sigma Aldrich, Merck, and Fisher Scientific standards. The quality requirement of the analytical grade NaCl was showed in Table 1.

Table 1 The quality of requirements for analytical grade NaCl

Parameter	Specification (%)
NaCl	99.50-100.50 *)
Magnesium and alkaline-earth metals as Ca	< 0.01
Sulfate	< 0.001
Calcium	< 0.002
Magnesium	< 0.001
Potassium	< 0.005
Water content	0.50

Notes: *) The NaCl content was calculated by determining chloride ions using an argentometric volumetric. Thus, the total measured chloride ions are from those bound with sodium and impurities ions.

This process produces analytical-grade salt. Therefore, the purification stages are necessary to increase NaCl content and reduce impurities of ions such as sulfate, calcium, magnesium, and potassium. Therefore, many studies have been conducted to improve the quality of salt by using several methods. Two of the common processes for purifying crystalline chemical compounds including salt is using the crystallization (Lu *et al*., 2017; Jones, 2002; Giulietti *et al*., 2001; Mullin, 2001) and precipitation methods (Mersmann, 2004).

These reported methods include re-crystallization (Rismana and Nizar, 2014; Rositawati, Taslim, and Soetrisnanto, 2013; Nurhidayati, 2007), hydro extraction (Kharismanto *et al.,* 2021; Sumada, Dewati, and Suprihatin, 2018; Martina *et al.,* 2016), coagulation (Astuti, 2021; Lukum *et al.,* 2021 Pujiastuti *et al.,* 2017; Rathnayaka *et al.,* 2014; 2013; Sulistyaningsih, Sugiyo, and Sedyawati, 2010; Ihsan and Djaeni, 2002; Yang, Yun, and Yang, 2007), geomembrane (Hoiriyah, 2019), purification – evaporation (Luo *et al.,* 2022; Schimdt, Meirhofer, and Schwaiger, 2009; Wae, Tjahjanto, and Mardiana, 2021), simultaneous crystallization (Penha, Zago, and Seckler, 2020; Zago, Penha, and Seckle, 2019; Penha *et al.,* 2018), purification using porous material composite (Sulhadi, *et al.,* 2017) and High-Density Polyethylene (HDPE) membrane (Rusiyanto Soesilowati, and Jumaeri 2013). However, these results are only able to produce industrial and food-grade salts.

The study of the process of purification of raw salt is evaluated by washing with a saturated solution of pure NaCl to remove the impurities. The results showed that washing the salt with brine can decrease contaminants such as magnesium, calcium, sulfate, and heavy metal (Ahhmed *et al.,* 2018). Furthermore, research to improve NaCl quality has been performed by drying the salt using a hot air-drying system. As a result, the NaCl concentration increased but could not reduce the impurity content (Amir *et al.,* 2021). Research for preparing analytical grade salt was investigated by re-dissolved, coagulation, ion exchange, and re-crystallization processes from rock salt (Rahman, Islam, and Farrukh, 2010). However, this studied process uses resin to reduce potassium levels, which is costly when developed on an industrial scale. The preparation of pharmaceutical-grade salt was also investigated using purification by precipitation and only one partially crystallized stage from crude salt as raw material (Rismana, 2016; Rismana and Srijanto, 2013). Nevertheless, the product still has a high potassium content which does not meet the NaCl analytical grade standard.

The novelty of this study was to reduce calcium, magnesium, and sulfate impurities using two stages of precipitation. Three processes of partial re-crystallization were also carried out to decrease potassium content. In addition, this production process produces a high-quality salt solution, which can be used as raw material to make pharmaceutical or food salts. Therefore, developing the process production technology analytical grade NaCl in Indonesia is still necessary at a laboratory and bench scale. The benefit of this research is the availability of the technology process and accessibility that the domestic industry can easily apply.

2. Materials and Methods

2.1. Materials

The materials are divided into raw, refining, and analysis groups. Raw and refining materials consist of crude salt, industrial-grade of Na_2CO_3 (PT. Brataco, Bandung, Indonesia), industrial-grade of NaOH and 32% HCl (PT Asahi Mas, Cilegon Indonesia), industrial-grade of $BaCl_2$ (Haihang Industry, Co. Ltd., Jinan, China). While, the materials pro analysis grade for analysis include $AgNO_3$, NaCl K_2CrO_4, Na-EDTA, EBT indicator, murexide indicator, 36% w/v HCl, NaOH, blue thymol bromine indicator, NH_4OH, NH_4Cl, Na_2SO_4, $BaCl_2$, oxalic acid (Merck, Germany).

2.2. Purification and Production Processes of Analytical Grade NaCl

The purification stages for this process will be used as follows: (1) crude salt was dissolved with water to obtain a brine solution with NaCl content of about 27-30 % w/v, (2) the brine solution was then filtered to remove insoluble impurities, (3) analysis of NaCl, SO_4^{2-}, Mg^{2+}, Ca^{2+}, water-insoluble content, and water content is carried out on the sample of the initial brine solution, the brine solution after purification, after filtering stages, and the analytical grade NaCl product, (4) purification and filtering process, (5) acidification and neutralization, (6) crystallization, and (7) separation and drying process. Figure 1 shows a complete set of production processes.

The purification process was performed using the precipitation of SO_4^{2-} with $BaCl_2$, Mg^{2+} with NaOH, and Ca^{2+} with Na_2CO_3 using the two-stage method. The first process was examined by coagulating SO_4^{2-} and Mg^{2+} ions using 20 % w/v $BaCl_2$ and NaOH solution successively, and after 12 hours left, the mixture was separated by the filtering process. The second process was studied by precipitation of Ca^{2+} ion using 20 % w/v Na_2CO_3 solution, and after 12 hours, the mixture was separated by the filtering process again. The detailed procedure was as follows; the brine solution was reacted with a 20% w/v $BaCl_2$ solution until the SO_4^{2-} the content was < 0.001 % (10 ppm). Furthermore, a brine solution containing $BaSO_{4(s)}$ was added 20% w/v NaOH to Mg^{2+} content of < 0.001 % (10 ppm). The next step was to separate $BaSO_{4(s)}$ and Mg $(OH)_{2(s)}$ by filtering.

The brine solution from the first stage is then continued to the second stage by reacting with 20% w/v Na_2CO_3 solution until the excess Ca^{2+} level < 0.001 % (10 ppm), and then separation is carried out. The brine solution's pH resulting from the second stage's separation was then reduced by acidification process using 1:1 HCl solution to pH 2-3. The pH was then raised by neutralization using 20% w/v NaOH solution to pH 5-6, followed by crystallization of pure brine solution. This process was carried out as many as three times. Finally, the salt product is separated from the high-quality brine solution and dried at 100-105°C.

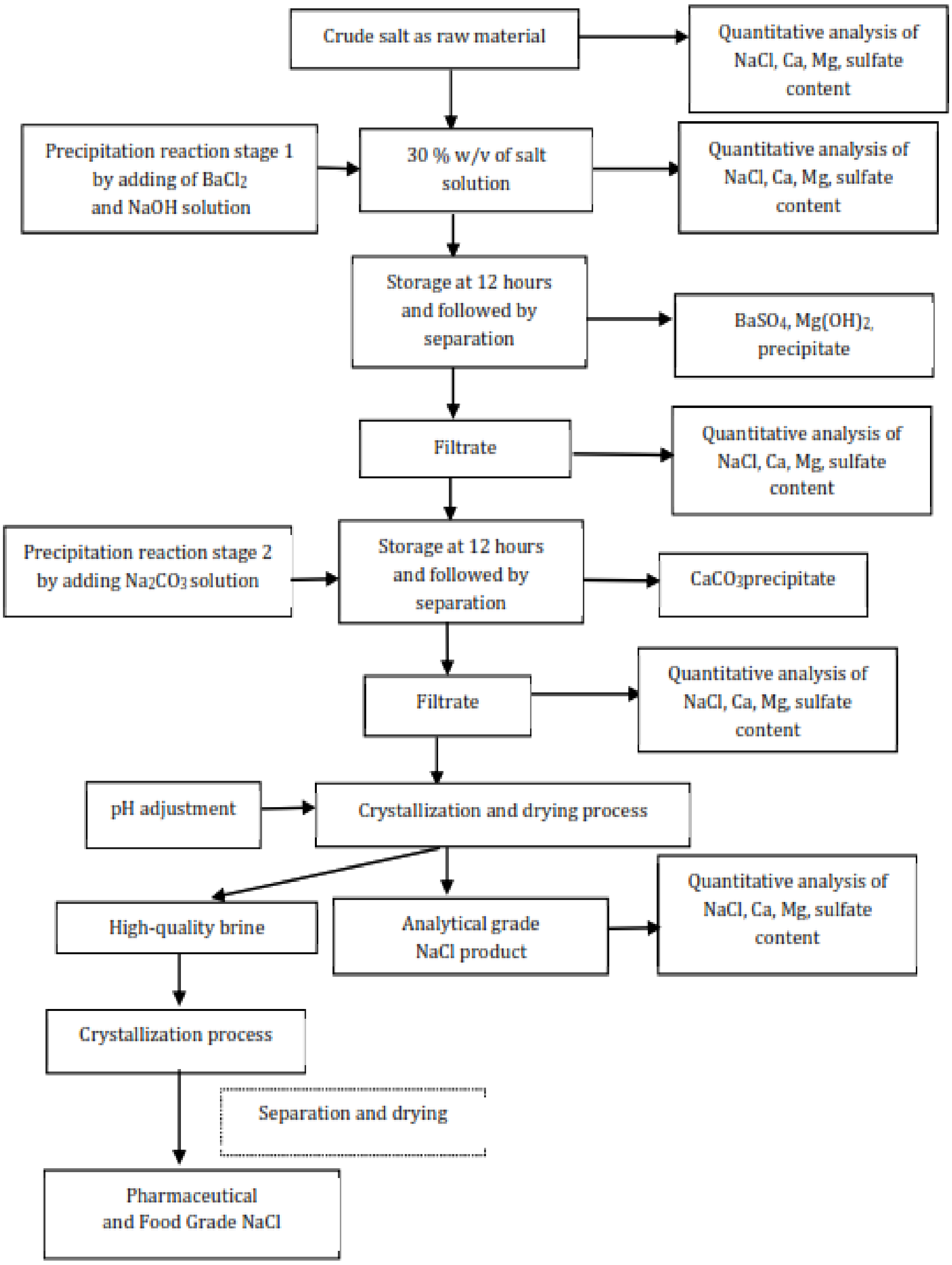

Figure 1 Schematic diagram of process production analytical grade NaCl by two stages of precipitation

2.3. Product Characterizations and Analysis

Product and intermediate NaCl were investigated according to the standard CoA of NaCl analytical grade. The NaCl content was calculated based on the chloride ion content determined by argentometry volumetric. While the calcium, magnesium, potassium, and heavy metal ions content were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) spectrophotometer. A pH solution was determined using a pH meter, and the Loss on Drying (LOD) apparatus measured water content. While sulfate content was determined using a turbidimetry spectrophotometer.

3. Results and Discussion

3.1. Analysis of Salt Raw Materials and 27-30 % w/v Initial Brine Solution

The quality of salt raw material needs to be analyzed to determine whether it meets the specification requirements. Table 2 shows the results of the analysis of NaCl content and the main impurities of the salt raw material. The analysis results in Table 2 show that raw salt materials have 87.30 % NaCl content. This salt indicates that the raw material still has low NaCl content. However, the calcium, magnesium, and sulfate impurities content is standard and meets consumption salt specifications. The content of water-insoluble impurities is relatively low; it will facilitate the separation of contaminants during the filtering process to get a clear salt solution.

Table 2 The results of the analysis of the raw materials salt

Parameter	Unit	Test Results
Shape, color, taste, and appearance	-	the crystal's size in millimeters (mm), cube-shaped, dull-white, salty
Solubility	-	Soluble in water, a small part of water-insoluble content, turbid salt solution
Identification	-	Positive on the sodium and chloride ion test
NaCl	%	87.30
Calcium (Ca^{2+})	%	0.31
Magnesium (Mg^{2+})	%	0.08
Sulfate (SO_4^{2-})	%	1.20
Barium	qualitative	Negative on the barium ion test
Water content	%	7.45
Insoluble water content	%	1.50

The higher NaCl and lower impurities compound content in salt raw material will be advantageous for producing industrial-grade salt (Chlor Alkali Plant (CAP), pharmaceutical, and analytical grade) due to fewer materials needed for refining and higher-yield products. The salt is dissolved in water to obtain a brine solution with a NaCl concentration of about 27-30% w/v. Adding water to the 27-30% salt solution also causes lower levels of calcium, magnesium, and sulfate impurities. Table 3 shows the analysis results of several main parameters of the initial brine solution.

Table 3 Analysis results of 27-30% w/v initial brine solution

Parameter	Unit	Test Results
Clarity of solution in water	-	The solution is turbid; a small part of the water-insoluble content
Identification test	-	Positive on the sodium and chloride ion test
NaCl	%	27.00
Calcium (Ca^{2+})	%	0.11
Magnesium (Mg^{2+})	%	0.03
Sulfate (SO_4^{2-})	%	0.35
Barium (Ba^{2+})	qualitative	Negative on the barium ion test
pH	-	7.00

3.2. Production of Brine Solution after Purification and NaCl Analytical Grade

Analyzing impurity ions in the brine solution is essential to determine the number of chemicals needed during refining. Table 4 and Figure 2 show the results of the NaCl content analysis and the brine solution's main impurities after purification. In this research, the purification process was carried out in two stages: the first stage of precipitation using 20 % w/v $BaCl_2$ and NaOH to reduce the sulfate (SO_4^{2-}) and magnesium (Mg^{2+}) ions content. In contrast, to reduce calcium (Ca^{2+}) ion content, we studied the second coagulation stage by adding 20 % w/v Na_2CO_3 to reduce the calcium (Ca^{2+}) ion content.

In the first stage, the amount of SO_4^{2-} and Mg^{2+} ions were decreased after precipitation with 20 % w/v $BaCl_2$ and NaOH at concentrations 0.0005-0.0006 % (5-6 ppm) and 0.0008-0.0012 % (8-12 ppm), respectively. The Ca^{2+} content did not significantly reduce because calcium did not react with NaOH and $BaCl_2$. After storage for 12 hours and filtration, the SO_4^{2-} and Mg^{2+} ions content remained relatively unchanged.

In the second precipitation stage, a reaction with 20 % w/v Na_2CO_3 reduced the concentration of Ca^{2+} ion to 0.0015 % (15 ppm). In contrast, SO_4^{2-} and Mg^{2+} ion content after 12 hours of storage and filtering showed a similar concentration. The NaCl concentration decreased from 26.30 % to 25.55 % after the first and second stage processes due to the dilution process of adding reagents. As presented below in Table 4, replicating three batches of the purification process showed good repeatability.

Table 4 Analysis of the result of brine solution after purification

Sample	Parameter	Batch 1	Batch 2	Batch 3	Average	SD
Brine solution after first-stage precipitation, storage at 12 h, and filtration	SO_4^{2-} (ppm)	5	5.5	7	5.83	1.04
	Ca^{2+}(ppm)	890	900	875	888.33	12.58
	Mg^{2+}(ppm)	8	12	9	9.67	2.08
	NaCl (%)	26.5	26.0	26.5	26.33	2.89
Brine solution after second-stage precipitation, storage at 12 h, and filtration	SO_4^{2-} (ppm)	5	5.5	7	5.83	1.04
	Ca^{2+}(ppm)	10	20	15	15.00	5.00
	Mg^{2+}(ppm)	5	6	6	5.67	0.58
	NaCl (%)	25.5	25.0	25.8	25.43	4.04

In the following process, HCl and NaOH solutions were added to control the pH brine solution in the acidification and neutralization stage. First, HCl solution is added, so the brine solution's pH becomes 2-3. Furthermore, the pH was raised again to pH 5-6 by adding a solution of NaOH. Adjusting the pH brine solution is essential to remove carbonate content so the product can meet the standard acidity specifications. Therefore, decreasing the pH to 2-3 in the salt solution was done to remove residual carbonate ions that should not be present in the NaCl pro analysis. While increasing the pH after that process to pH 5-6 is to return the pH of the solution to be neutral so that the final NaCl product can meet the pH requirements of 6-7 when testing the pH of the solution. challenging.

The crystallization process stage evaporates 50-60% of the water from a brine solution. The crystallization process of the high brine solution is not performed thoroughly to keep the potassium ions from crystallizing and can result in the NaCl analytical grade product being unable to pass the requirements. The process was repeated three times so that the potassium level remained less than 0.0050 % (50 ppm). However, high brine solution still contains high salt content and low impurity to be used as raw material for pharmaceutical and food salt production.

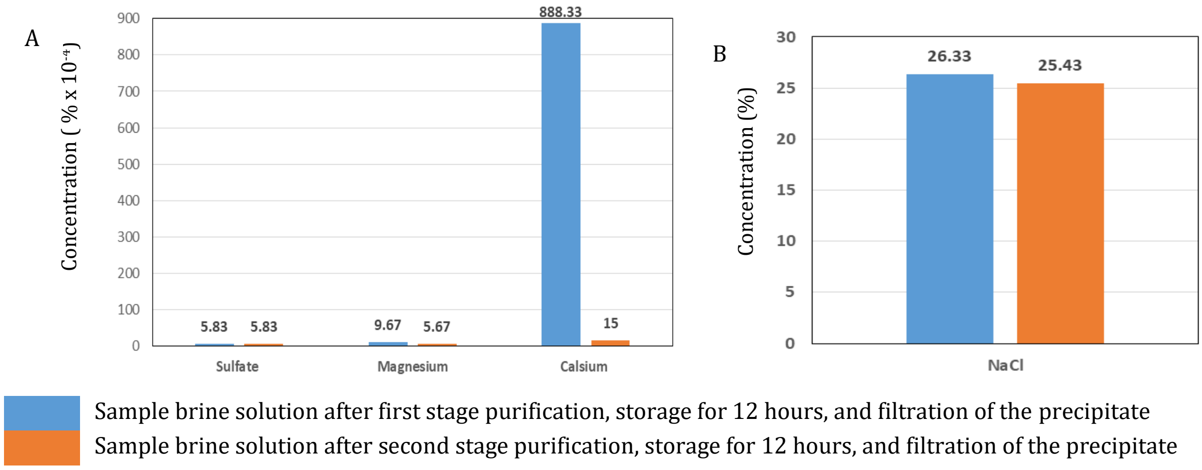

Figure 2 A. Content of sulfate, calcium, magnesium, and B. Content of NaCl after purification by two stages of precipitation

3.3. Production of Analytical Grade NaCl Product

The process was validated by a triplicate. Table 5 presents the yield and analysis of product results. The average yield of the process production was about 30.33 %. Meanwhile, the quality of the product also met the requirements of the main parameters of the salt analytical grade specifications. In addition to calcium, magnesium, sulfate, and potassium impurities, barium must also be tested as impurities because it is a heavy metal and one of the reagents added as $BaCl_2$ to meet the requirements according to the standard.

Table 5 Analysis results of quality of analytical grade NaCl

Parameter	Batch 1	Batch 2	Batch 3	Average	SD
Yield, %	30.00	32.00	29.00	30.33	1.53
NaCl, %	100.50	100.10	99.85	100.15	0.33
Ca^{2+}, %	0.0010	0.0012	0.0009	0.0010	0.0002
Mg^{2+}, %	0.0009	0.0010	0.0010	0.0010	0.0001
SO_4^{2-}, %	0.0008	0.0005	0.0007	0.0007	0.0002
pH	6.00	6.00	7.00	6.33	0.58
H_2O,%	0.40	0.45	0.50	0.45	0.05
K^+, %	0.0050	0.0049	0.0040	0.0046	0.0006
Arsen	pass	pass	pass	-	-
Cuprum	pass	pass	pass	-	-
Plumbum	pass	pass	pass	-	-
Ferrum	pass	pass	pass	-	-
Barium	pass	pass	pass	-	-

One advantage of this process is that the outcome by-products of high-quality brine solution can be used as a raw material for producing pharmaceutical and food-grade salts. Table 6 shows the analysis of pharmaceutical-grade salt after crystallization and drying from high-quality residual brine. The pharmaceutical-grade salt product offers good quality with NaCl, Ca^{2+}, SO_4^{2-} and Mg^{2+} contents and meets the requirements set by pharmacopeia.

Table 6 Analysis of results of pharmaceutical-grade salt from the high-quality brine solution

Test parameter	Batch 1	Batch 2	Batch 3
NaCl, %	99.70	99.75	99.00
Ca^{2+} and Mg^{2+}, %	0.0028	0.0030	0.0035
SO_4^{2-}, %	< 0.02	< 0.02	< 0.02
K^+, %	< 0.05	< 0.05	< 0.05
Water content, %	0.40	0.45	0.45
pH	6.00	7.00	6.50
Arsen	pass	pass	pass
Cuprum	pass	pass	pass
Plumbum	pass	pass	pass
Ferrum	pass	pass	pass
Barium	pass	pass	pass

4. Conclusions

This research carried out the salt production process through purification, acidification, neutralization, crystallization, separation, and drying methods. This developed technology process of analytical grade salt production at the laboratory scale has produced salt according to the specifications required, with an average product yield of 30.33 % and a purity of NaCl of 100.15 %. Its simplicity and ability to produce accompanying products. Compared to existing technologies, the advantages of this production technology are its simplicity and ability to make multiple accompanying products. Moreover, by-products of high-quality brine in this technology can be used as raw material to produce pharmaceutical and food-grade salt. Therefore, this invention has excellent potential to accommodate domestic demands, substitute imported products, and increase the economic value of crude salt as raw material.

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