SYNTHESIS OF FOOD GRADE SALT FROM BRINE WATER OF TIRTA SANITA BOGOR

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ABSTRACT

Tirta Sanita Bogor brine water contains high concentrations of sodium (Na). It also contains calcium (Ca) and magnesium (Mg). The purpose of this research is to make the food grade salt with relatively low Ca and Mg content. The process was carried out in two methods, the first method was chemical precipitation which was divided into two stages, the removal of the Mg element using a limestone compound (CaCO_3) which has been calcined at 900°C for 6 hours, and the removal of Ca element using Li_2CO_3 compound. The second method was traditional evaporation which was conducted using prism greenhouse technology. The brine filtrate was reacted with Na_2CO_3 and the results were analyzed using the ICP-OES. The results showed that the NaCl content was 80% by the chemical precipitation and traditional evaporation methods.

Keywords: Brine water, Precipitation, Evaporation, Food grade salt

INTRODUCTION

Indonesia has a number of beaches with the overall area of about 54,716 km². These beaches stretch across the archipelago from Sabang to Papua. However, the long coast does not guarantee that it is able to meet the needs of domestic salt production. This is due to a short dry season (4 to 5 months), high humidity (60-70%), traditional salt production equipment and methods. As a result, the salt quality is relatively low (88-92.5%).

Salt is a chemical compound called sodium chloride (NaCl). This compound is hygroscopic, so that it can not be obtained in completely pure form. According to the Ministry of Trade, salt is a compound which main component consists of sodium chloride (NaCl) and contains other compounds such as water, magnesium, calcium, and sulfate ions. Based on its utilization, salt is classified into two groups, namely table salt and industrial salt. According to SNI, table salt has a minimum NaCl content of 95% (Tansil et al., 2016). For industrial salt, better quality is highly needed, for example, in the petroleum industry, the NaCl content is 97% (Rismana, 2014) the textile and leather tanning industry requires NaCl content above 97.5%, the chlorine alkaline industry requires NaCl above 98.5% and the pharmaceutical industry requires above 99.5%. The need for salt in the chemical industry sector is increased significantly every year, therefore the purification of local salt should be grown in order to meet market needs. Table 1 shows the parameter of salt quality based on SNI 01-3556-2000 standards (Rusiyanto et al., 2013).

According to (Yansa et al., 2015) the classification of NaCl salts has several criteria, such as "very good" with more than 95% of NaCl content, "good" criteria, with the NaCl content between 90 and 95%, and "moderate" criteria, with the percentage from 80 to 90%.

Brine water of Tirta Sanita Bogor contained high concentrations of NaCl nevertheless, still contained Ca and Mg of 38.48 and 146.02 ppm respectively (Lalasari et al., 2019). This is the reason why Tirta Sanita Bogor brine water has potency to produce industrial salt with food grade quality. The aim of this research is to study the salt from brine water of Tirta Sanita Bogor from non-food grade to food grade quality salt. The processing was carried out in two

methods, the first method was used chemical precipitation and the second method was applied the traditional evaporation.

Table 1. The parameter of salt quality

Compound	Content
Sodium Chloride, NaCl	min. 94.7%
Water	max. 5%
Iodine as KIO ₃	30-80 mg/kg
Calcium and Magnesium	1.00%
Sulfate, SO ₄ ²⁻	2.00%

Source: (Rusiyanto et al., 2013)

The first method of chemical precipitation included: the process of removing magnesium (Mg) using 150% excess limestone (CaO) which has been calcined at 900°C for 6 hours (Firdiyono et al., 2020). The Mg removal reaction was conducted by the formation mechanism of Mg(OH)₂ precipitates and followed by filtration. The second stage was the removal of the calcium (Ca) using the Li₂CO₃ therefore, the CaCO₃ precipitates was formed and removed Mg by the formation of MgCO₃ precipitates simultaneously. Brine filtrate which was expected to be nearly free from Mg and Ca was reacted with Na₂CO₃ (Sumarno, Ratnawati & Nugroho 2012) to obtain Na salt. The second method of traditional evaporation was executed by using prism greenhouse technology, the salt was washed and separated from impurities and the result was compared with the salt from the literature.

METHODOLOGY

Materials

The materials used are shown in Table 2.

Table 2: The list of chemicals

Materials	Specification
Brine Water	Brine water of Tirta Sanita Bogor
CaCO ₃	Rembang Limestone (Technical)
Li ₂ CO ₃	Merck, solid (99.997%)
Na ₂ CO ₃	Merck, solid (99.99%)
Distilled water	Clear

Equipment:

The equipment used consist of

a) Reflux equipment includes:

1) Stative rod 2) clamp, 3) Condenser, 4) Thermometer, 5) Three neck flask, 6) Cool water bath 7) Magnetic stirrer, 8) Hotplate and Magnetic Stirrer.

b) Filtration equipment (filtering) consist of:

1) Stative rod 2) clamp 9) Whatman filter paper No. 41, 10) Separating Funnel, and 11) Beaker Glass.

Figure 1.(a) Reflux equipment set and (b) Filtrating equipment set (Lalasari dkk, 2019)

Research design:

Method 1 – Preparation of Na salts by chemical precipitation

- Raw materials preparation, including the filtering of brine water from the prism greenhouse.
- Chemical Precipitation 1 (removal process of Mg)
- Amount of 482.44 grams of CaO was added to 4 L of brine water and then continued by filtration
- Next, stirring was performed for 3 hours by magnetic stirrer
- After that, the filtration was conducted. The results was called filtrate A.
- Chemical Precipitation 2 (removal process of Ca)
- Amount of 10 mL of filtrate was taken from filtrate A and then it was added Li_2CO_3 with variation of 0, 50, 100 and 150%.
- After that, It was refluxed at 30°C . for 4 hours.
- Furthermore, the filtration was conducted (Figure 1 b). The result was called filtrate B which was tested by ICP-OES.
- Finally, amount of 50 mL $\text{Na}_2\text{CO}_3 \cdot 12\text{H}_2\text{O}$ was added into 1 Liter of filtrate B to form Na salt.

Method 2 – Production of Na salt by Traditional Evaporation

- Salt was collected from prism greenhouses
- After that, It was filtered to clean from impurities. Furthermore, the separation was carried out based on the hexagonal salt form.
- Eventually, the salt resulted was compared with its structure in the literature.

The research diagram is illustrated in Figure 2.

Method 1

Method 2

Process of Making Na salt

Figure 2. The flowchart of Food Grade Salt production

Research Stages

The research stage was carried out by two methods:

Method I

Chemical precipitation was performed by removing the magnesium (Mg) using excess of 150% limestone (CaO) which has been calcined at 900°C for 6 hours. (Firdiyono dkk, 2020). The Mg removal reaction was done by the precipitation of Mg(OH)₂ and followed by filtration. The second stage was the removal of calcium (Ca) using the Li₂CO₃. The CaCO₃ precipitate

was formed and simultaneously removed the remaining Mg with the formation of MgCO_3 precipitate. The Brine filtrate was expected to be free from Mg and Ca by Na_2CO_3 as reactant to obtain Na salt (Sumarno et al., 2012).

Method II

The formation of Na salts with traditional evaporation was conducted by using the prism greenhouse technology (a technology using rectangular building with a geoisolator base and a prism-shaped plastic roof). The salt was obtained from harvesting process. After that, washing process was conducted in order to separate the impurity and followed by crystallization. The salt that has been formed was then compared with salt from the literature.

Analysis Procedures

The product analysis in this study was the salt content test using the Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) instrument. The analysis was performed in Metallurgy and Materials Research Center - LIPI, Puspitek, Serpong.

RESULTS AND DISCUSSION

1. The formation of Na salts by Using Chemical Precipitation Method

Based on the research of Lalasari et al., (2019), the content of Brine water of Tirta Sanita Bogor contained Ca, Mg, Li and Na components with concentration of 384.866, 147.227, 746.748 and 877.891 ppm respectively. According to (Aprilianti & Gustiawati, 2016) study, the content of brine, Na has had range from 25.000 to 100.000 ppm, Mg and Ca was about 57.000 and 75.000 ppm respectively. Based on the first precipitation (Mg removal), filtrate A was based on the following reaction

The test results with the ICP-OES instrument are shown in Table 3.

Table 3. Results of Phase -1 Precipitation with ICP-OES Test

Element	Concentration (ppm)
Mg	18.290
Ca	820.136
Li	951.973
Na	782.965

The analysis results in table 3 shows that the concentration of Mg has decreased to 18.290 ppm, (reaction 2). Mg content in the previous study was 209.353 ppm since some of the Mg has formed $\text{Mg(OH)}_{2(s)}$ precipitate. Thus, this shows that the remaining Mg content in the brine filtrate is around 8.740%.

The results of filtrate B in reaction stage II (Ca removal) are based on the following reactions:

This stage was carried out with a variation of Li_2CO_3 (excess seeding) at 0, 50, 100 and 150%, which was refluxed at 30°C for 4 hours. The test results are shown in Figure 3.

Figure 3. Effect of the Excess on Ca Removal at 30°C and 4 Hours

Based on the graph in Figure 3, it can be seen that the removal of Ca at the temperature of 30°C for 4 hours is 99.60%, i.e the remaining Ca is 0.640 %. This is because part of the filtrate has formed $\text{CaCO}_{3(s)}$. Moreover, some of the remaining Mg left in the filtrate has also settled into $\text{MgCO}_{3(s)}$. Therefore, there is still Ca content by 0.640% in the brine filtrate. After the reduction in the Ca content at reaction 3 and 4, there is still Lithium at temperature of 150°C with the concentration of 1746 ppm. Figure 4 shows the lithium content in the reduction process of Ca content.

Figure 4. Effect of the Excess on Li Concentration at 30°C and for 4 Hours

It can be seen in Figure 4 that the Li concentration in the Brine filtrate is 1746 ppm. This indicates that the Lithium content in the 1-liter filtrate is 1746 mg. On the other hand, Na salt can be obtained from the following reaction:

According to the reaction 5 above, it means that the Na salt formed is equivalent to the amount of Lithium salt in amount of 1746 grams. At temperature of 30°C for 4 hours, the Li concentration becomes 1,373.02 mg. Thus, the percentage of Na salt obtained was 78.72%. Furthermore, the portion of Mg and Ca was 8.74 and 0.64% respectively by the chemical precipitation

2. The Formation of Na salt by Traditional Evaporation Method

Study to the salt resulted by harvesting which was conducted using a prism greenhouse has already done. The salt was analyzed after the purification from its impurities. There are several forms of Na salt images, as shown in Figure 5.

Figure 5. Several forms of Salt; a) Salt obtained from prism greenhouse harvest, b) Na salt resulted from separation process of prism greenhouse harvest, c) Na salt from literature, d) Na salt produced from recrystallization

Based on Figure 5 above, in Figure a) It can be seen different form of salt. This is proved that the salt also contains not only Na but also Ca and Mg as impurities (Arwiyah et al., 2015). Meanwhile, in figure b) shows the Na salt which has been purified from its impurities. It has cube-shaped Na salt therefore it can be said that this is Na salt resulted from the harvesting of the prism greenhouse. Furthermore, there is a similarity of shape when compared to the salt in figure c) and d), which belongs to the group of medium class of NaCl salts (Yansa et al., 2015).

By comparing between Na salt from traditional evaporation and in Figure d), it is almost similar. Thus, it can be said that Na salt obtained by chemical precipitation is almost similar with Na salt resulted from traditional evaporation. Nevertheless, in terms of health, salt resulted from traditional evaporation is considered more healthier for human since it does not use any chemicals. According to the standard for the quality of salt based on the SNI 01-3556-2000 (Rusiyanto et al., 2013), the category of food grade salt has 94.7% of NaCl content which similar to study by (Yansa et al., 2015) which has NaCl content from 90 to 95%. On the other hand, the medium class salt has 80% of NaCl (Farikh, 2017). Therefore, in this study, the salt resulted by using chemical precipitation and traditional evaporation has food grade salt quality with 80% of NaCl and been classified in the medium grade salt.

CONCLUSION

The results showed that food grade salt from brine water of Tirta Sanita Bogor by chemical precipitation and evaporation is classified in “medium” grade salt with 80% of NaCl content.

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