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PRODUCTION OF PURIFIED SODIUM CHLORIDE AS INDUSTRIAL STANDARD

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ABSTRACT

The research described about chemical treatments to increase percentage of sodium chloride from crude salt which was obtained from four districts on Madura Island. The conventional salt process contains percentage of sodium chloride 85.99%; 81.90%; 84.825% and 86.87% from Sumenep, Pamekasan, Sampang and Bangkalan respectively. Those results were still below the standard quality of industrial salt (98.5% of dry base). In this study, increasing percentage of sodium chloride was done by re-crystallization method. Crude salt was diluted with distilled water to form saturated solution, then added sodium hydroxide, barium chloride and hydrochloride acid to remove impurities such as magnesium hydroxide, calcium hydroxide, potassium hydroxide, barium sulfate, and carbonic acid. The results showed that the purified of sodium chloride enhanced 100.03%; 98.86%; 98.86% and 99.7% for salt products from Sumenep, Pamekasan, Sampang and Bangkalan, respectively and accepted by industrial standard.

Keywords: *Purified salt, chemical treatments, industrial standard*

INTRODUCTION

Madura is one of the largest salt producing islands in Indonesia. Approximately 15,000 hectares of salt land are located in Madura; therefore, Madura earned the title as "Salt Island". However, this great potential does not coincide with an increase in the quantity and quality of salt. Traditional salt is produced by solar evaporation of seawater in the crystallization ponds and influenced significantly by raw materials, production processes and post-harvest salt management systems. Most of traditional salt in Madura is still below standard quality of salt industry. The crude salt contents sodium chloride (NaCl) 80%-90% and impurities such as Magnesium, calcium, potassium, sulphate and carbonic ions (Rathnayaka, 2014).

Purified sodium chloride is a major commodity needed as a raw material in the chlor-alkali industry by electrolysis of a salt solution to produce chlorine gas, hydrogen gas, sodium hydroxide and potassium hydroxide. The content NaCl of industrials salt must be above 98.5 % (dry based) (Seville,2000). Production of purified sodium chloride as industrial standard can be achieved by chemical treatments to bind the impurities then separated from sodium chloride easily. This study aims increasing concentration of NaCl using three settler processes in stage using sodium hydroxide (NaOH), barium chloride ($BaCl_2$) and hydrochloride acid (HCl). Theoretically, based on metathesis reaction and solubility value of compounds, the adding NaOH, $BaCl_2$ and HCl in saturated solution of crude salt can precipitate magnesium hydroxide, calcium hydroxide, barium sulphate and release carbonic acid in gas. Consequently, concentration of sodium chloride will improve as follows the reactions:

$MgCl_{2(aq)} + NaOH_{(aq)} \rightarrow Mg(OH)_{2(s)} + NaCl_{(aq)}$
$CaCO_{3(aq)} + NaOH_{(aq)} \rightarrow Ca(OH)_{2(s)} + Na_2CO_{3(aq)}$
$BaCl_{2(aq)} + Na_2SO_{4(aq)} \rightarrow BaSO_{4(s)} + 2NaCl_{(aq)}$
$2HCl_{(aq)} + Na_2CO_{3\,(aq)} \rightarrow H_2CO_{3(g)} + 2NaCl_{(s)}$

(Ihsan & Djaeni, 2002; Pujiastuti, 2017).

MATERIALS AND METHODS

This study was done in salt laboratory, University of Trunojoyo Madura and the crude salt was obtained from four districts in Madura.

The sodium chloride analysis was conducted by titration methods (SNI-8207-2016). Sodium hydroxide, barium chloride and hydrochloride acid as precipitation agents and all other chemicals used in this research were analytical grade. Schematically, the production of purified sodium chloride was shown in following **Figure 1**.

The modified methods of Pujiastuti (2017) and Ihsan & Djaeni, (2002) were used to production of purified sodium chloride as industrial standard. The first purification was treated by 60 ml sodium hydroxide 2 mol in 1L saturated solution of crude salt which filtered before by Whatman paper. This solution was stirred then the sediment was filter. The filtrate was crystallized with moisture content maximum 5 % and analyzed sodium chloride. The second purification was conducted by 15 ml Barium chloride 0.1 mol in 1L saturated salt produced from previous step. This solution was stirred then the sediment was filter. The filtrate was crystallized with moisture content maximum 5 % and analyzed sodium chloride. Furthermore, the third purification was treated by 10 ml hydrochloride acid 0.2 mol in 1L of saturated salt produced from the second purification. This solution was stirred and heated to release carbonic acid. The filtrate was crystallized with moisture content maximum 5 % and analyzed sodium chloride.

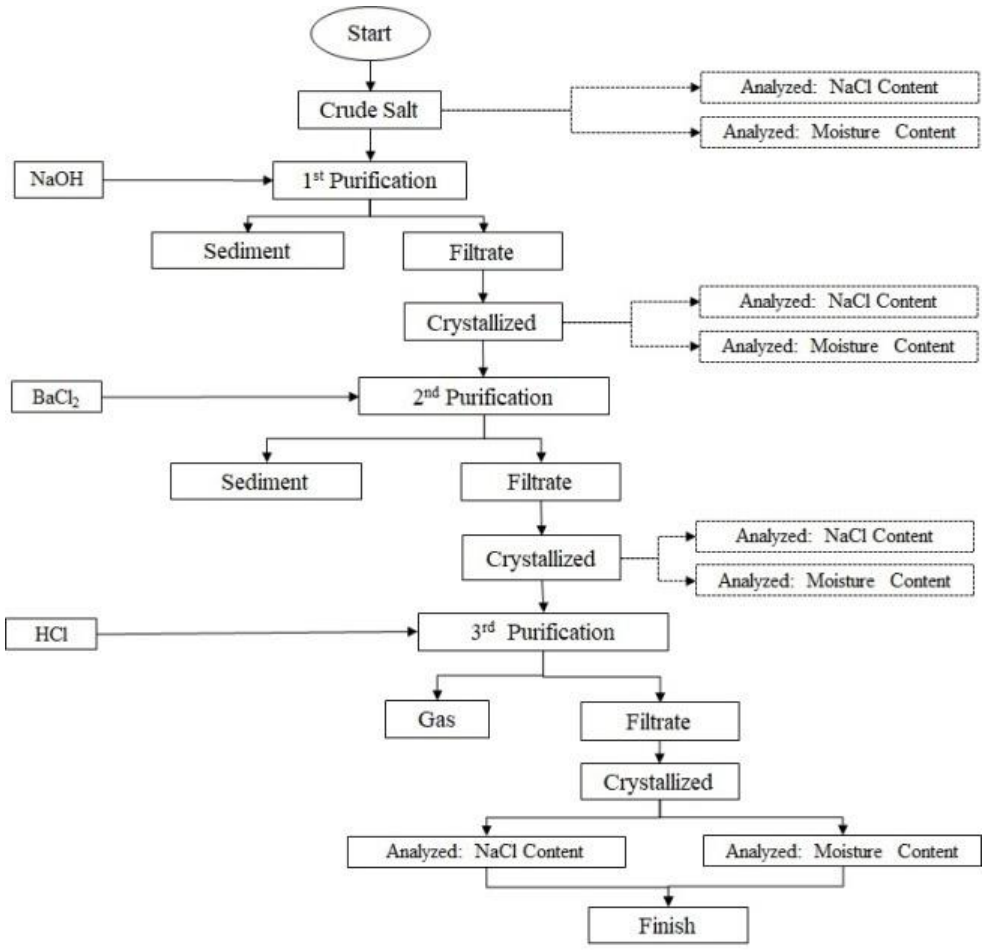

Figure 1. Schematic Diagram of research

RESULT AND DISCUSSION

The concentration of sodium chloride in crude salt from Sumenep, Pamekasan, Sampang and Bangkalan were showed in **Figure 2**. The NaCl concentrations were 85.99%, 81.90%, 84.825% and 86.87% for crude salt products from Sumenep, Pamekasan, Sampang and Bangkalan respectively. **Figure 3** was presented physical properties of crude salt in Madura. The appearance of crude salt was dirty and it was not proper to consume even as the raw material of industry.

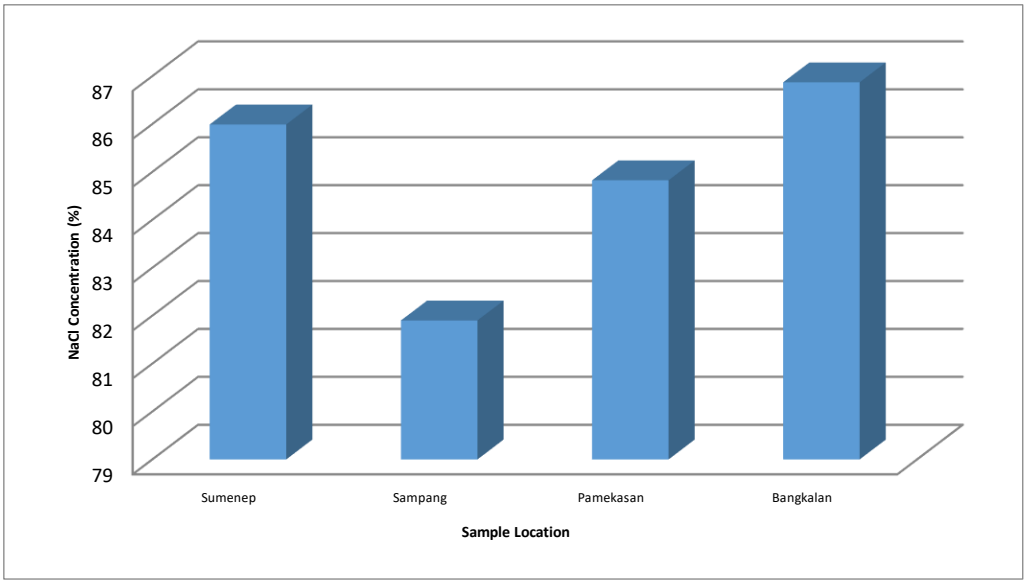

Figure 2. Concentration of Sodium chloride in crude salt

Figure 3. Appearance of crude salt

Concentration of Sodium Chloride after the first purification

The first purification is purpose to reduce magnesium and calcium ion. Based on solubility data showed solubility of magnesium hydroxide and calcium hydroxide was lower than sodium chloride (Pujiasuti, 2017). Hence, the metathesis reaction between crude salt with sodium hydroxide can increase the concentration of sodium chloride, as below the reactions:

$MgCl_{2(aq)} + NaOH_{(aq)} \rightarrow Mg(OH)_{2(s)} + NaCl_{(aq)}$
$MgSO_{4(aq)} + NaOH_{(aq)} \rightarrow Mg(OH)_{2(s)} + Na_2SO_{4(aq)}$
$CaCO_{3(aq)} + NaOH_{(aq)} \rightarrow Ca(OH)_{2(s)} + Na_2CO_{3(aq)}$

Sodium carbonic (Na_2CO_3) and sodium sulphate (Na_2SO_4) were treated in the further step to reduce these impurities. **Figure 4** expressed that salt after first purification from four districts contains higher concentration of sodium chloride than crude salt. **Figure 3** showed that the NaCl concentrations were 95.062%, 91,26%, 93,6% and 95.35 % for the first purification of salt from Sumenep, Pamekasan, Sampang and Bangkalan respectively. The results increase around 9% from concentration of sodium chloride in crude salt. **Figure 7b** showed appearance of salt in this step was whiter and refined than crude salt.

Pudjiastuti *et al.* (2017) explained that the addition of sodium hydroxide solution is more effective in removal of magnesium ions than calcium ions. Ihsan & Djaeni, (2002) also reported that adding NaOH 2 grams in public salt solution can precipitate 0.94 g of magnesium hydroxide.

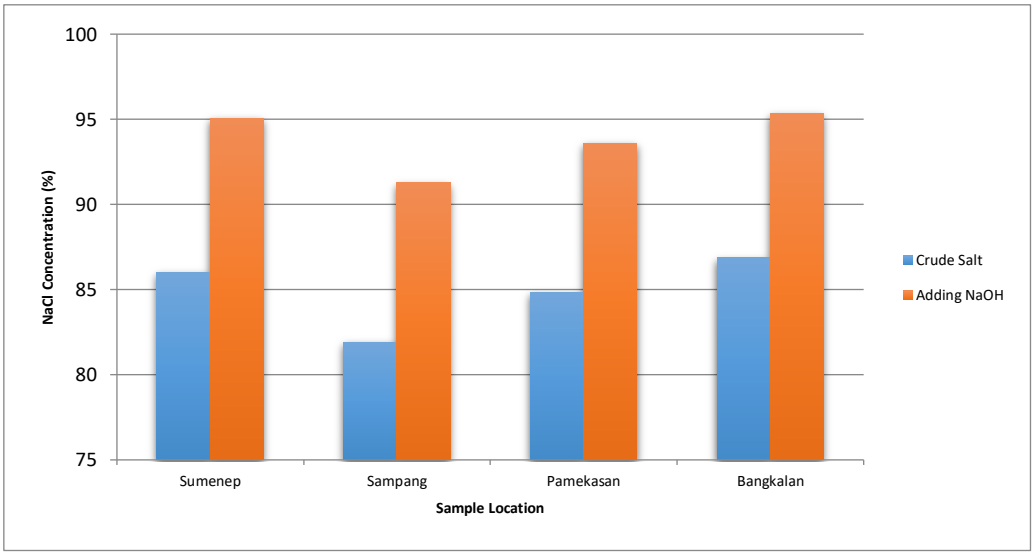

Figure 4. Concentration of sodium chloride after the first purification

Concentration of Sodium Chloride after the second purification

The second purification aims to reduce sulphate ions after adding barium chloride to form barium sulphate as the below complete reactions:

$BaCl_{2(aq)} + Na_2SO_{4(aq)} \rightarrow BaSO_{4(s)} + 2NaCl_{(aq)}$

Sodium sulphate as impurities produced from the previous step can be removed by adding barium chloride. Moreover, this method can increase concentration of sodium chloride in sample. **Figure 5** showed that concentrations of sodium chloride of salt after this step were 97.69%, 94,185%, 97.11% and 97.4% for salt from Sumenep, pamekasan, Sampang and Bangkalan respectively. The percentage of sodium chloride increases around 3 % from previous step. **Figure 7c** presented appearance of 2nd purification was better than before. Ihsan & Djaeni, (2002) explained the addition 2.5 grams barium chloride can precipitate 2.5 grams of barium sulphate.

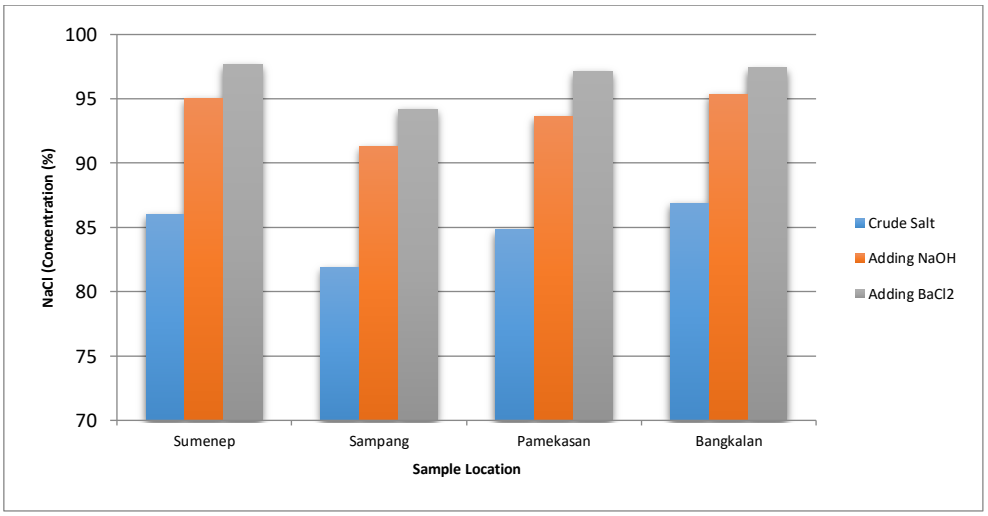

Figure 5. Concentration of sodium chloride after the second purification

Concentration of Sodium Chloride after the third purification

The third purification aims to release gas carbonic acid by adding hydrochloride acid as the below reaction:

$CaCO_{3(aq)} + NaOH_{(aq)} \rightarrow Ca(OH)_{2(s)} + Na_2CO_{3(aq)}$

$2HCl_{(aq)} + Na_2CO_{3\,(aq)} \rightarrow H_2CO_{3(g)} + 2NaCl_{(s)}$

The sodium carbonic as impurities produced from the second purification can be removed by adding hydrochloride acid. **Figure 6** showed that concentration of sodium chloride accepted industrial standard (NaCl > 98.5%). Sodium chloride content after this step obtained 100.03%, 98,86%, 98,86% and 99,7% for salt

products from Sumenep, Pamekasan, Sampang and Bangkalan respectively. These data were compared with Indonesian national standards for industrial salt SNI 8207:2016 whereby NaCl contents obtained were reached above 97%. Besides that, **Figure 7d** showed salt appearance of this method was the best result.

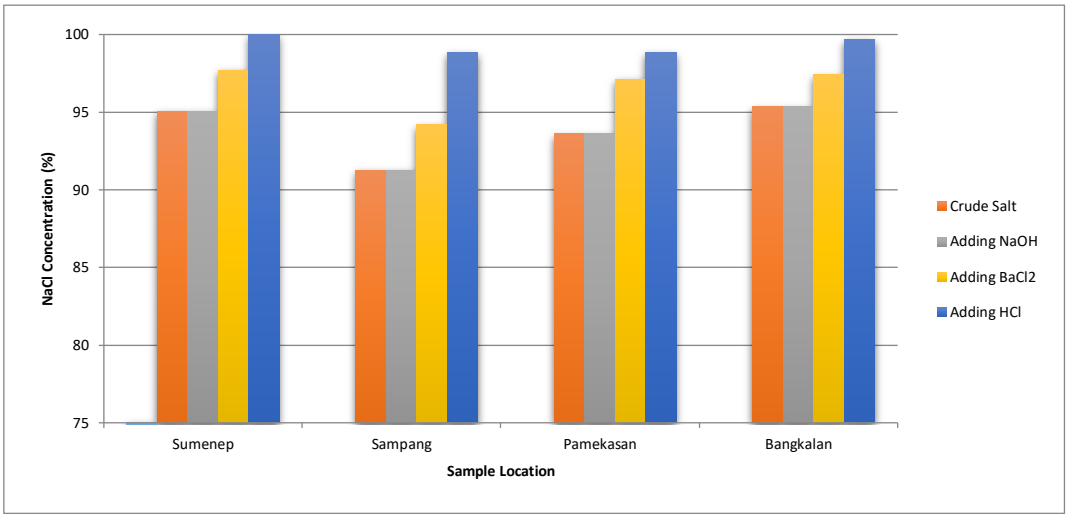

Figure 6. Concentration of sodium chloride after the second purification

Figure 7. Comparison of salt appearance: (a) crude salt; (b) 1st purification; (c) 2nd purification and (d) 3rd purification

CONCLUSIONS AND SUGESTION

In this study, we can increase of sodium chloride percentage as industrial standard by using sodium hydroxide 2 mol, Barium chloride 1 mol and hydrochloride acid 2 moles. The purified of sodium chloride enhanced 100.03%; 98.86%; 98.86% and 99.7% for salt products from Sumenep, Pamekasan, Sampang and Bangkalan, respectively.

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