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Synthesis of Sodium Hydroxide from Traditional Salt Through Electrolysis Process

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A B S T R A C T

Indonesia is expected to compete with other countries in the industrial sector, which plays a crucial role in the economic growth of a nation for its development. In several industries in Indonesia, NaOH plays a critical role in the production process, serving both as a primary component and as a supporting component. In the chemical industry, the chlor-alkali process is a crucial electrolytic technique utilised. The products generated from this process include Cl_2, H_2, and NaOH. This research utilizes traditional salt as a raw material for the production of NaOH, considering that traditional salt has a high NaCl content and also contains H_2O, which is expected to yield NaOH with optimal concentration. This study aims to produce technical-grade NaOH that complies with the Indonesian National Standard (SNI) by examining the effects of electrical voltage and electrolysis time on the NaOH produced through the electrolysis process. The stages of this research include the preparation of salt solution, the electrolysis process, and product analysis. The study was conducted with a NaCl concentration of 86.72% at electrical voltages of 5, 7.5, 10, 12.5, and 15 volts during the electrolysis process. Additionally, the electrolysis durations used were 100, 150, 200, 250, and 300 minutes. The weight of NaOH produced at an electrical voltage of 12.5 volts and a time of 100 minutes was 1.081 grams of NaOH

Contribution to Sustainable Development Goals (SDGs)
SDG 9- Industry, Innovation, and Infrastructure
SDG 12- Responsible Consumption and Production

1. INTRODUCTION

1.1. Research Background

In the industrial sector, Indonesia is anticipated to compete with other nations. The industrial sector is essential for a country's economic growth and development. Sodium hydroxide (NaOH) is essential in various Indonesian industries, serving as both a primary and auxiliary raw ingredient in manufacturing processes. Now, Indonesia's need for NaOH is predominantly met through imports. Data from the Central Statistics Agency indicates that Indonesia imported 55,037.75 tons of NaOH per year. In 2021, Indonesia imported 54,985.1 tons of sodium hydroxide. In 2022, the import volume rose to 94,386.39 tons annually. In 2023, Indonesia imported 74,462.71 tons of NaOH. This research will focus on the manufacture of NaOH in response to the steadily rising annual demand for NaOH. This study used community salt as a precursor for NaOH synthesis, given its elevated NaCl concentration and the presence of H_2O, which is anticipated to produce NaOH with optimal concentration. The chlor-alkali process is a crucial electrolysis method in the chemical industry. The products produced from this procedure comprise Cl_2, H_2, and NaOH, derived from the electrolysis of NaCl solution. To obtain chlorine and NaOH as final products, the electrolysis cell must be configured to prevent the mixing of these two chemicals. The electrolysis process for creating NaOH is affected by various parameters, including reactant concentration. The reaction rate will increase with the increasing concentration of the reactant solution. An additional significant aspect is the electrical voltage;

an increase in voltage applied results in an accelerated reaction rate, and conversely [1].

1.2. *Literature Review*

1.2.1. Traditional Salt

Cations, or positive ions, and anions, or negative ions, make up the ionic compound known as salt. Reactions between bases and acids can result in the creation of salt. Organic molecules like chloride (Cl^-) and acetate (CH_3COO^-), monatomic ions like fluoride (F^-), or polyatomic ions like sulfate (SO_4^{2-}) can all be the source of the cations and anions in salt [2]. Seawater evaporates and crystallizes to create community salt, sometimes referred to as crude solar salt. Sodium chloride (NaCl) and a variety of other dissolved salts, the majority of which are impurities comprising ions like Ca^{2+}, Mg^{2}+, and SO_4^{2-}, are typically present in the salt produced by Indonesian salt farmers. These ions typically bond to form compounds such as calcium sulfate ($CaSO_4$), magnesium sulfate ($MgSO_4$), and magnesium chloride ($MgCl_2$), as well as other compounds [3].

Table 1. Composition of traditional salt

No	Component	Unit	Content
1	Water	%	6.06
2	NaCl	%	86.34
3	Magnesium (Mg)	%	0.104496
4	Cadmium (Cd)	$Mg.kg^{-1}$	0.58
5	Lead (Pb)	$Mg.kg^{-1}$	8.23
6	Copper (Cu)	$Mg.kg^{-1}$	1.47
7	Mercury (Hg)	%	0.0001

1.2.2. Sodium Hydroxide (NaOH)

Caustic soda, also known as sodium hydroxide (NaOH), is one type of metal that possesses caustic properties. When sodium oxide, a basic oxide, dissolves in water, it produces sodium hydroxide. Caustic soda, or NaOH, dissolves to form a solution with elevated alkalinity. During the electrolysis process, hydroxide ions (OH^-) and sodium ions (Na^+) can react at the cathode to generate NaOH through a reaction mechanism. In conjunction with other ions, the Na^+ ions generated by the plasma's effect on the dissociation of NaCl at the anode migrate towards the cathode (negative electrode). As NaOH is a strong base, its synthesis transpires after the arrival of Na^+ ions at the cathode. The ions interact with the hydroxide ions (OH^-) in the solution, leading to the synthesis of a new molecule, NaOH [4].

1.2.3. Electrolysis

Electrolysis is a chemical process that transforms electrical energy into chemical energy. The electrodes and electrolytes are the two primary elements in this process. The core principle of electrolysis contrasts with that of a voltaic cell, as the reactions in electrolysis are non-spontaneous. This procedure necessitates electrical energy from an external source and occurs within an electrolytic cell, which comprises an electrolyte solution or molten electrolyte and includes two distinct types of electrodes. The electrolysis process decomposes water molecules into their constituent atoms by transmitting an electric current across two electrode poles. The cathode, linked to the negative terminal, possesses a negative charge, whereas the anode, associated with the positive terminal, possesses a positive charge. Electrodes are conductors utilized for establishing contact with either a component or a non-metallic material within a circuit, such as an electrolyte solution or a semiconductor. An electrochemical cell comprises many electrodes, which are metallic conductors submerged in an ionic conductive electrolyte. This electrolyte may exist as a solution, solid, or combination. The electrodes and electrolyte delineate the configuration of the electrode chambers. Graphite is an inert substance that serves as a highly efficient electrocatalyst and exhibits resistance to diverse solution conditions. The selection of graphite electrodes depends on multiple aspects, including cost, accessibility of raw materials from diverse natural sources, simplicity of synthesis, and the capacity to get it in powder, fiber, or composite forms. Graphite exhibits a hexagonal crystal structure, characterized by a structural foundation of graphene sheets or carbon layers [5].

1.2.4 Eletrodialysis

Transferring ions from one solution to another via an ion exchange membrane while being affected by an electromagnetic field is known as electrodialysis. Electrodialysis entails the migration of ions via an anion and cation-selective membrane from a less concentrated solution to a more concentrated solution, driven by the passage of a direct electric current. The ions in the solution can traverse the ion exchange membrane as a result of the electric field's impetus. The exchange membrane serves to safeguard the oxidative anolytes from oxidising the product and intermingling with the catholytes, while also retaining the cation product [6].

1.3. *Research Objective*

This study seeks to generate technical-grade sodium hydroxide (NaOH) following the SNI 0074-2011 standard, examining the influence of applied voltage and electrolysis time on NaOH synthesis through the electrolytic process.

2. MATERIALS AND METHODS

2.1 *Materials and Tools*

The primary raw material used in this study is traditional salt obtained from the salt ponds in Lembung Village, Galis District, Pamekasan, Madura. The supporting raw material for this research is distilled water (aquadest) sourced from Nirwana Sains UD in Surabaya, East Java, which will be utilized as a solvent. The apparatus for the electrolysis process consists of a power supply, electrodes, and a membrane.

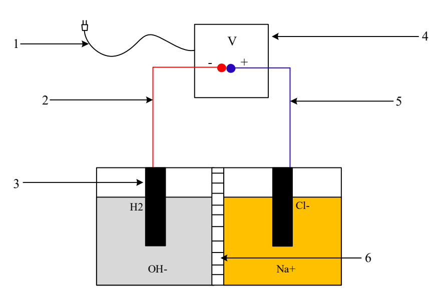

Figure 1. Electrolysis Equipment

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Description :

1. Electricity
2. Cathode (-)
3. Elektrode (grafit)
4. *Power Supply*
5. Anode (+)
6. Cathode exchange membrane

2.2 Research Procedure

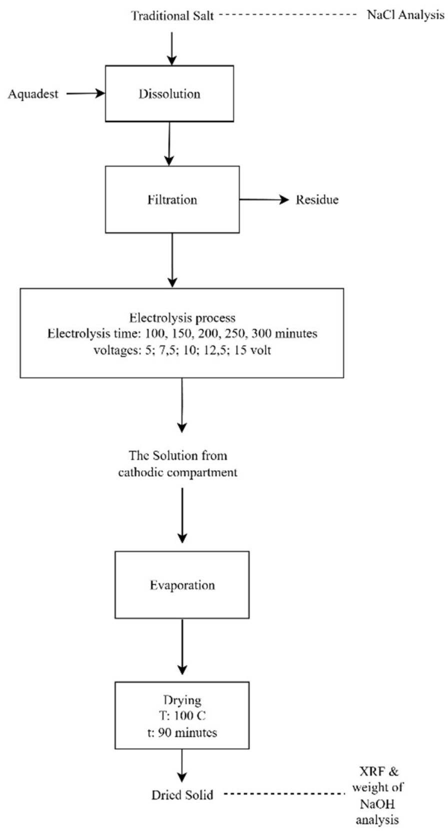

Figure 2. Procedure

The traditional salt to be used is first analyzed to determine its NaCl content. Subsequently, the analyzed traditional salt is dissolved in 500 mL of distilled water (aquadest). The salt solution is then filtered using filter paper. The filtered salt solution is introduced into the anode compartment, while distilled water is added to the cathode compartment. Following this, the electrolysis process is carried out according to the predetermined variables. A solid will form in the cathodic compartment. The solution in the cathodic compartment is evaporated to reduce the water content. An oven is used to dry the resulting solid for 90 minutes at a temperature of 100ºC. The dried solid is weighed using an analytical balance. Next, the dried solid is analyzed using XRF testing to determine the Na content present in the solid.

After determining the Na content from the XRF test, the analysis of NaOH weight is conducted based on the obtained Na content using the following formulas :

- Calculating Na Weight = Sample Weight x % Na
- Calculating NaOH Weight = Na Weight x (Molecular Weight of NaOH) / (Atomic Weight of Na)

3. RESULT AND DISCUSSION

3.1. Traditional Salt Analysis

In the conducted research, traditional salt was used as the raw material for the production of NaOH through a specific process. The obtained traditional salt was then analyzed at the Testing and Calibration Laboratory of the National Standardization Agency and Industrial Service Policy using Titrimetric and Gravimetric methods. The purpose of this testing is to measure the NaCl content present in the traditional salt, resulting in the following analysis:

Table 2. Analysis of NaCl Content in Traditional Salt

No	Parameter	Unit	Result	Test Method
1	Sodium Chloride (NaCl) adbb	%	87.72	Titrimetry
2	Sodium Chloride (NaCl) adbk	%	95.9	Titrimetry
3	Water Content (H_2O)	%	8.53	Gravimetry

Based on the NaCl test results, the Na+ ion content in the traditional salt can be analyzed. The calculations indicate that with a NaCl content of 87.72%, the Na+ ion content is 34.47%.

3.2 The Correlation between Electrical Voltage and Weight of NaOH

The filtered salt solution, with its NaCl content determined, was then subjected to electrolysis using varying electric voltage and predetermined time intervals. The results of this study yielded a fluctuating graph. The graph illustrating the effect of electric voltage on the weight of NaOH is shown in Figure 3.

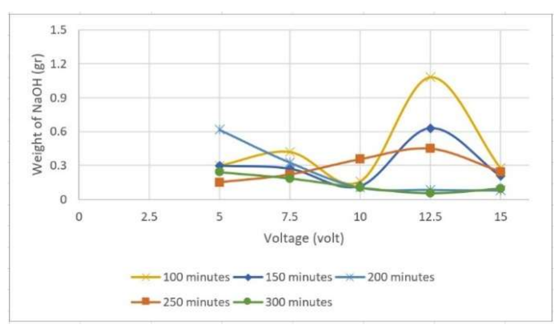

Figure 3. Correlation between Electrical Voltage and Weight of NaOH

It is known that as the voltage increases, the reaction rate also increases. A higher electric voltage allows for more ions to participate in the conduction of electric current. The increased number of ions involved in the conduction of electricity contributes to a higher reaction rate [7]. However, the results of this study indicate that at certain voltage levels, the weight of NaOH tends to decrease or stagnate, even at higher voltages. This may be due to several factors, such as the efficiency of the electrolysis cell, solution saturation, or the presence of side reactions that hinder NaOH formation. At high voltages, an increase in solution temperature can occur, causing some electrical energy to convert into heat. Consequently, a significant portion of the energy used is wasted as heat rather than breaking water bonds, leading to energy loss and decreased efficiency. Thus, while increasing voltage does enhance electrolysis results, this effect only persists up to a certain point before the efficiency of NaOH formation declines due to side reactions or overheating [8]. Side reactions can occur at high voltages, resulting in the formation of by-products that can reduce efficiency [9]. Furthermore, at higher voltages, graphite experiences corrosion due to the degradation of carbon by oxygen into carbon dioxide, which impairs the graphite's ability to conduct electricity optimally [10].

3.3 The Correlation between Electrolysis Time and Weight of NaOH

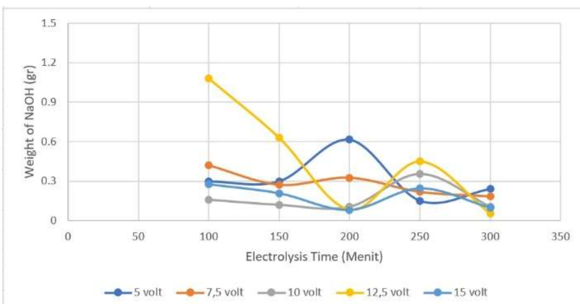

Figure 4. Correlation between Electrolysis Time and Weight of NaOH

Figure 4 illustrates the relationship between the electrolysis time on the weight of NaOH. This study yielded a fluctuating graph. At the beginning of the electrolysis process, the weight of NaOH produced tends to be high because the process is still operating optimally, and the ions are available in sufficient quantities. However, as the electrolysis time increases, the solution becomes saturated, causing the formed Na+ and OH- ions to no longer accumulate as NaOH, or reverse reactions may occur that reduce NaOH yield. Prolonged electrolysis time can lead to decreased efficiency due to side reactions or water evaporation [8]. For instance, at a voltage of 12.5 volts, the weight of NaOH sharply increases at the 100th minute but then drastically decreases in the following minutes, indicating the occurrence of reverse reactions or loss of efficiency. At voltages of 5 and 15 volts, the weight of NaOH also fluctuates, demonstrating that excessive time does not necessarily yield greater results. Extended durations can lead to increased energy consumption without a significant increase in NaOH yield. Electrolysis time increases, reaction efficiency decreases due to the accumulation of gas bubbles (H_2 and Cl_2) on the electrodes, which hinders ion contact with the electrode surface, thereby reducing the reaction rate for NaOH formation [11]. This results in the NaOH concentration not increasing with prolonged time. Extended electrolysis time also leads to the formation of more corrosive free chlorine, resulting in a decrease in electric current voltage or the applied voltage in the apparatus not aligning with the expected voltage. This corrosive free chlorine can impair performance [12].

3.4 Characterisation and Implementation of NaOH

Sodium hydroxide produced from the electrolysis of salt solution (NaCl) using membrane cells is known as membrane-grade or technical-grade sodium hydroxide. This type of sodium hydroxide is the primary product of the chlor-alkali process. The physical characteristics of NaOH obtained from electrolysis include a clear liquid solution with an initial NaOH concentration of approximately 30–35%, which can be further processed through evaporation to achieve a concentration of 50% NaOH. This NaOH is highly corrosive, readily soluble in water, and has a very high pH. The solid NaOH obtained can be in the form of flakes, pearls, or pellets. In this study, the solid NaOH was obtained in powder form, although the particle size was not uniform, necessitating a screening process to meet industry standards. Membrane-grade caustic soda is more energy-efficient and environmentally friendly compared to products from diaphragm or mercury cell processes [13]. The standard for caustic soda (NaOH) is SNI 0074-2011, which regulates quality specifications for both solid and liquid caustic soda. The standard parameters for solid NaOH require a concentration of $\geq$98% and NaCl content of $\leq$0.1%. Based on these parameters, the NaOH produced in our study does not yet comply with the SNI 0074-2011 standard.

Caustic soda (NaOH) can be used as a primary or supporting raw material in various industries. In the chemical industry, NaOH is used as a material for producing sodium salts. Additionally, in the paper industry, NaOH catalyzes dissolving lignin during the pulping process, thereby accelerating the separation and breaking of fibers. In the textile industry, NaOH is also an important material used to enhance tensile strength, absorbency, and luster in cotton fabrics, and it can also be utilized during the bleaching process to improve bleaching efficiency [13].

4. CONCLUSION

Based on the data obtained from this study, the following conclusions can be drawn:

1. The effect of electric voltage on the weight of NaOH produced is directly proportional. As the electric voltage during the electrolysis process increases, the weight of NaOH obtained also increases. However, at excessively high voltages, an increase in solution temperature may occur, causing some electrical energy to convert into heat, thereby reducing the energy available for breaking water bonds and leading to side reactions that decrease the efficiency of NaOH formation. Therefore, it is essential to observe the optimal operating conditions to ensure effective processing. In this study, the optimal voltage for NaOH formation was found to be 12.5 volts with a duration of 100 minutes, yielding a NaOH weight of 1.081 grams.
2. The effect of electrolysis time on the weight of NaOH is also directly proportional. The longer the electrolysis process is conducted, the greater the weight of NaOH produced. However, prolonged electrolysis can lead to decreased efficiency due to electrode degradation, side reactions, or consumption of OH^- ions by other reactions. Additionally, the solution may become saturated, hindering NaOH

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formation and resulting in a reduced yield. In this study, the optimal electrolysis time was determined to be 100 minutes at a voltage of 12.5 volts.

3. NaOH produced from electrolysis using membrane cells is classified as membrane-grade or technical-grade NaOH, which can be in the form of flakes, pearls, or pellets, per the SNI 0074-2011 standard. However, the NaOH produced in this study did not meet this standard. According to SNI 0074-2011, the standard for NaOH is ≥98%. In our research, the highest NaOH concentration was obtained at a voltage of 12.5 volts and an electrolysis time of 150 minutes, yielding a NaOH concentration of 2.81%, a solid weight of 22.39%, and a NaOH weight of 0.630 grams.

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