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# Production of Calcium Nitrate Fertilizer from Calcium Hydroxide Waste: Effects of Reaction Temperature and Nitric Acid Concentration

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## ABSTRACT

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) waste generated from the acetylene gas industry contains a high calcium content and has potential to be utilized as an alternative raw material for calcium nitrate fertilizer production. This study presents a novel approach for the valorization of  $\text{Ca}(\text{OH})_2$  industrial waste into a value-added calcium nitrate fertilizer through a reaction and crystallization process. The effects of reaction temperature and nitric acid concentration on calcium nitrate production were investigated. The  $\text{Ca}(\text{OH})_2$  waste was initially reduced to a particle size of 100 mesh, then reacted with nitric acid at concentrations of 1, 1.25, 1.5, 1.75, and 2 N and at reaction temperatures of 50, 60, 70, 80, and 90°C for 1 hour. The obtained calcium nitrate solution was filtered, neutralized using 25%  $\text{NH}_4\text{OH}$  until pH 7, and crystallized at 150°C to obtain calcium nitrate crystals. The raw material was characterized using X-ray fluorescence (XRF), while the calcium nitrate product was analyzed using inductively coupled plasma (ICP), spectrophotometry, and yield calculation. The XRF analysis showed that  $\text{Ca}(\text{OH})_2$  waste contained 98.71% calcium. At a reaction temperature of 70°C and a nitric acid concentration of 1.5 N, calcium nitrate was successfully produced with the highest calcium (Ca) and nitrogen (N) content of 21.91% and 16.74%, respectively, and produced a yield of 69.564%, which is in accordance with the Indonesian National Standard (SNI 02-2806-1992) for calcium nitrate fertilizer, which stipulates a Ca content of 19–22% and an N content of 15.5%. This study was conducted without experimental replication; therefore, statistical significance analysis was not performed. The findings demonstrate that  $\text{Ca}(\text{OH})_2$  waste from the acetylene industry can be effectively valorized into calcium nitrate fertilizer, providing an alternative strategy for industrial waste utilization and supporting circular economy principles.

### Contribution to Sustainable Development Goals (SDGs):

**SDG 2: Zero Hunger**

**SDG 8: Decent Work and Economic Growth**

**SDG 9: Industry, Innovation, and Infrastructure**

**SDG 12: Responsible Consumption and Production**

**SDG 13: Climate Action**

## 1. INTRODUCTION

### 1.1. Research Background

Acetylene industrial waste is a by-product generated from the acetylene gas industry. The production of  $\text{Ca}(\text{OH})_2$  waste is

estimated to reach approximately 1,370 million tons per year, making it the third-largest industrial by-product in the world. This waste, commonly known as carbide waste or calcium hydroxide waste, is produced from the reaction between calcium carbide and water, which generates acetylene gas ( $\text{C}_2\text{H}_2$ ) as the main product and calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) as the by-product. Acetylene gas is commonly used for welding and combustion processes,



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while the resulting  $\text{Ca(OH)}_2$  waste still contains a high calcium content and has potential for further utilization [1]. Calcium hydroxide waste can be utilized in various applications, such as acid waste neutralization, fertilizer production, soil stabilization [2], and as an alternative raw material for clinker production in the cement industry [3], thereby providing added value and reducing the risk of environmental pollution. In addition, acetylene gas waste reportedly contains up to 91.7% calcium [4].  $\text{Ca(OH)}_2$  waste is regarded as a viable alternative source for calcium nitrate fertilizer manufacturing because of its substantial calcium content. The utilization of this waste not only increases the economic value of industrial by-products but also supports waste valorization into useful and value-added products, particularly in the agricultural sector [5].

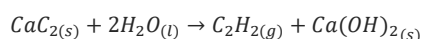
Research on the synthesis of calcium nitrate fertilizer was conducted by Pujahashita (2022) in a study entitled "Synthesis of  $\text{Ca(NO}_3)_2$  Fertilizer from Bleaching Earth Industrial Waste." This study utilized bleaching earth industrial waste, which was reacted with nitric acid ( $\text{HNO}_3$ ). The process conditions were varied by adjusting the  $\text{HNO}_3$  concentration at 0.8, 1.0, 1.2, 1.4, and 1.6 M, as well as reaction temperatures of 50, 55, 60, 65; and 70°C. At an  $\text{HNO}_3$  concentration of 1.6 M and a reaction temperature of 70°C, the process reached optimum conditions, with the synthesized product exhibiting 23.3% calcium (Ca) and 0.64% total nitrogen [6].

Although previous studies have demonstrated the feasibility of producing calcium nitrate fertilizer from industrial waste, most studies have focused on other waste sources, while the utilization of acetylene-derived  $\text{Ca(OH)}_2$  waste as a raw material for calcium nitrate fertilizer production remains limited. Previous applications of carbide residue have mainly focused on cement production and construction materials, whereas its potential as a fertilizer precursor has not been extensively investigated. Therefore, further study is needed to explore the conversion of  $\text{Ca(OH)}_2$  waste from the acetylene industry into calcium nitrate fertilizer and evaluate the effects of reaction parameters, particularly nitric acid concentration and reaction temperature, on product quality.

Based on the above description and considering previous studies, the researchers will produce calcium nitrate fertilizer from  $\text{Ca(OH)}_2$  waste by applying variations in temperature and nitric acid concentration through dissolution and crystallization processes in order to meet the Indonesian National Standard (SNI 02-2806-1992). The calcium nitrate production process involves reacting calcium hydroxide with nitric acid during a dissolution stage. Subsequently, the crystallization process is employed to obtain solid calcium nitrate fertilizer. This stage involves heating to remove the solvent from the liquid calcium nitrate solution, thereby forming solid crystalline products.

## 1.2. Literature Review

Calcium hydroxide ( $\text{Ca(OH)}_2$ ) is a waste generated from the industrial production of acetylene gas ( $\text{C}_2\text{H}_2$ ). The formation of this waste occurs through the interaction of calcium carbide ( $\text{CaC}_2$ ) and water ( $\text{H}_2\text{O}$ ), which primarily yields acetylene gas ( $\text{C}_2\text{H}_2$ ) and simultaneously produces calcium hydroxide ( $\text{Ca(OH)}_2$ ) as a by-product. The acetylene gas is separated in the gaseous phase, while  $\text{Ca(OH)}_2$  remains as a solid waste. The chemical reaction that produces this waste is reported by [7].



The properties of calcium hydroxide ( $\text{Ca(OH)}_2$ ) are presented in the following table [8]:

**Table 1.** Characteristic Calcium Hydroxide

| No | Specifications   | Description    |
|----|------------------|----------------|
| 1  | Physical form    | Solid          |
| 2  | Color            | Greyish-white  |
| 3  | Water Solubility | 0.185 g/100 mL |
| 4  | pH               | >12            |

Calcium nitrate fertilizer ( $\text{Ca(NO}_3)_2$ ) is classified as an inorganic fertilizer containing two essential nutrients, namely calcium (Ca) and nitrogen (N), which play an important role in plant growth. Calcium (Ca) and nitrogen (N) in calcium nitrate fertilizer work synergistically to support plant development [9]. Calcium strengthens plant cell walls, resulting in more rigid plant structures, improved tissue quality, and the prevention of physiological disorders such as blossom-end rot. Meanwhile, nitrogen in the nitrate form plays a key role in chlorophyll formation and supports the photosynthesis process, thereby promoting faster and optimal vegetative growth (leaves and stems) [10]. Therefore, calcium nitrate fertilizer not only enhances plant strength and structural quality through calcium (Ca), but also accelerates plant growth and productivity through nitrogen (N) [11].

The quality requirements of calcium nitrate fertilizer based on the Indonesian National Standard (SNI 02-2806-1992) are as follows [12]:

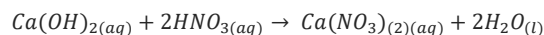
**Table 2.** SNI (Indonesian National Standard) specification for calcium nitrate fertilizer

| No | Specifications   | Description          |
|----|------------------|----------------------|
| 1  | Content          | 15.5 % N; 19-22 % Ca |
| 2  | pH               | 7                    |
| 3  | Water Solubility | 1200g/L              |

### 1.2.1. Process

#### A. Dissolution

Dissolution is a process in which two or more substances mix to form a homogeneous solution. An unsaturated solution contains a solute concentration that is still below the maximum solubility limit at a given temperature. A saturated solution occurs when the solute is in equilibrium with its solid phase. In contrast, a supersaturated solution contains a higher amount of solute than its solubility limit at a certain temperature [13]. The production of calcium nitrate fertilizer begins with dissolving calcium hydroxide ( $\text{Ca(OH)}_2$ ) waste in nitric acid ( $\text{HNO}_3$ ) to form a calcium nitrate solution.



Dissolution is conducted in a beaker glass under heating conditions with continuous stirring using a magnetic stirrer.

#### B. Crystallization

Crystallization is a process of crystal formation from a solution. The crystallization process aims to obtain a solid product with the desired purity and crystal size [14]. In the production of calcium nitrate fertilizer, the crystallization process begins with heating the solution at a temperature above 100°C to increase its concentration by reducing the water content. After reaching a

supersaturated condition, the solution is cooled to room temperature, allowing calcium nitrate crystals to start forming. There are three stages in the crystallization process, namely: (1) supersaturation; (2) nucleation; and (3) crystal growth, all of which are important for producing solid crystalline substances.

Supersaturation is a condition in which the concentration of a solute in a solution exceeds its solubility limit, thereby promoting crystal formation. Nucleation represents the initial stage of crystal nucleus formation from a supersaturated solution. These nuclei subsequently develop during the crystal growth stage through the addition of solute molecules or ions onto the crystal surface. Factors such as temperature, cooling rate, stirring, and solution concentration influence the size and shape of the resulting crystals [15].

### 1.2.2. Factors Affecting the Production of Calcium Nitrate Fertilizer

The production of calcium nitrate fertilizer is influenced by several factors during its manufacturing process. The factors affecting the process are as follows:

#### A. Temperature

Temperature significantly affects the purity and quality of the calcium nitrate produced. Excessively high temperatures may lead to the formation of undesirable by-products. In addition, the heating temperature also influences particle size and surface area of the resulting product [6].

#### B. pH

Improper pH conditions can affect the quality of the produced calcium nitrate. Maintaining a near-neutral pH promotes efficient calcium nitrate formation and minimizes undesirable side reactions. Previous studies have shown that pH 7 provides favorable conditions for achieving high and stable nitrogen content [16].

#### C. Stirring Time

Stirring is an important parameter in crystallization as it enhances mass transfer and influences nucleation and crystal growth, thereby affecting crystal size distribution and settling behavior [17].

#### D. Concentration

The concentration of reactants affects the reaction rate. Higher concentration results in a greater number of particles in the system, thereby increasing the probability of particle collisions. This leads to an increase in effective collisions, which in turn accelerates the conversion of reactants into products [18].

### 1.3. Research Objective

This study aims to produce calcium nitrate fertilizer utilizing acetylene gas waste in the form of  $\text{Ca}(\text{OH})_2$  through a crystallization process, as well as to investigate the effect of temperature and  $\text{HNO}_3$  concentration on the calcium (Ca) content and nitrogen (N) content of the obtained calcium nitrate fertilizer. The main objective of this study is to produce calcium nitrate fertilizer that meets the quality standards specified in SNI 02-2806-1992.

## 2. MATERIALS AND METHODS

The primary raw material used in this experimental study was  $\text{Ca}(\text{OH})_2$  waste from the acetylene gas industry. The additional materials used in the production of calcium nitrate fertilizer

include nitric acid ( $\text{HNO}_3$ ), distilled water ( $\text{H}_2\text{O}$ ), and 25% ammonium hydroxide ( $\text{NH}_4\text{OH}$ ). All materials were selected to support the formation and crystallization of calcium nitrate fertilizer. The equipment used in this study includes an analytical balance, a glass beaker, a glass stirring rod, a magnetic stirrer, a dropper pipette, a pH meter, a glass funnel, and filter paper.

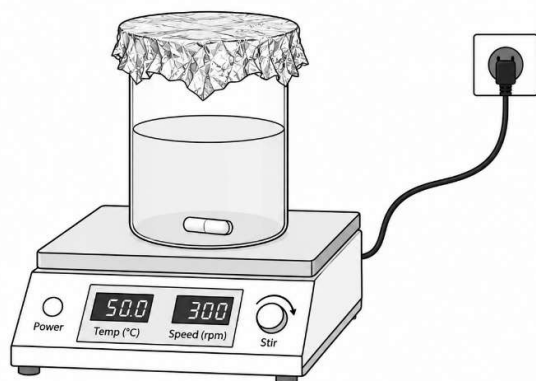
The production of calcium nitrate fertilizer in this study was conducted using fixed variables, including a  $\text{Ca}(\text{OH})_2$  waste mass of 40 g, 25%  $\text{NH}_4\text{OH}$  concentration, a stirring time of 1 hour, a stirring speed of 300 rpm, and a crystallization temperature of  $150^\circ\text{C}$ . The investigated variables in this study were dissolution temperature and  $\text{HNO}_3$  concentration. The reaction temperature was varied at  $50^\circ\text{C}$ ,  $60^\circ\text{C}$ ,  $70^\circ\text{C}$ ,  $80^\circ\text{C}$ , and  $90^\circ\text{C}$ , while the  $\text{HNO}_3$  concentration was varied at 1 N, 1.25 N, 1.5 N, 1.75 N, and 2 N, using 400 mL of  $\text{HNO}_3$  solution. These variations were determined based on a preliminary study to obtain calcium nitrate fertilizer with calcium (Ca) and nitrogen (N) contents that meet SNI standards. The experimental procedure began with grinding the  $\text{Ca}(\text{OH})_2$  waste, followed by sieving through a 100-mesh screen. The material was then weighed at 40 g. Subsequently,  $\text{Ca}(\text{OH})_2$  waste was dissolved in nitric acid ( $\text{HNO}_3$ ) in a beaker glass placed in an ice bath to control the temperature increase due to the exothermic reaction. The solution was then heated and stirred with a magnetic stirrer at the predetermined temperature for 1 hour. After the reaction stage, the mixture was first filtered to remove any insoluble impurities. The filtrate was then subjected to pH measurement using a pH meter, followed by neutralization through the addition of 25%  $\text{NH}_4\text{OH}$  until the pH reached 7 (neutral). The precipitate formed during the neutralization process was separated by filtration. The resulting calcium nitrate filtrate was then subjected to crystallization by heating at  $150^\circ\text{C}$  until water evaporation occurred, leading to the formation of white calcium nitrate crystals. The crystallization temperature of  $150^\circ\text{C}$  was selected to accelerate solvent evaporation and promote crystal formation. According to Zhao et al. (2022), calcium nitrate tetrahydrate undergoes stepwise dehydration within the temperature range of  $50\text{--}220^\circ\text{C}$ . Therefore, at  $150^\circ\text{C}$ , partial dehydration of hydrated calcium nitrate may occur. Since crystal phase characterization was not performed in this study, the hydration state and crystal structure of the final product could not be conclusively determined. Nevertheless, the temperature remained far below the decomposition temperature of calcium nitrate ( $>550^\circ\text{C}$ ), indicating that thermal decomposition of the nitrate compound was unlikely [19].

$\text{Ca}(\text{OH})_2$  waste was first characterized using X-Ray Fluorescence (XRF) analysis to determine its chemical composition and calcium content before being used in the synthesis process. The calcium nitrate product obtained was then characterized using Inductively Coupled Plasma (ICP) to determine calcium concentration and UV-Vis spectrophotometry to analyze nitrogen content, as well as yield calculations to evaluate the efficiency of the synthesis process. The results of the analysis were then compared with the requirements stipulated in the Indonesian National Standard (SNI 02-2806-1992) for calcium nitrate fertilizer.

The percentage yield of calcium nitrate was calculated using the following equation:

$$\text{Yield (\%)} = \frac{W_{\text{actual}}}{W_{\text{theoretical}}} \times 100\% \quad (1)$$

Wactual is the final dry weight of calcium nitrate obtained (grams), and Wtheoretical represents the theoretical mass of calcium nitrate calculated based on the stoichiometric conversion of  $\text{Ca(OH)}_2$  (grams). A total of 25 experimental conditions were evaluated by combining five reaction temperatures and five  $\text{HNO}_3$  concentrations. Each experimental condition was conducted once ( $n = 1$ ). Therefore, replicate measurements, error analysis, and statistical significance tests were not performed.



**Figure 1.** Experimental setup for the preparation of calcium nitrate

### 3. RESULT AND DISCUSSION

#### 3.1. XRF Analysis of $\text{Ca(OH)}_2$ Raw Material

Calcium hydroxide ( $\text{Ca(OH)}_2$ ) waste from the acetylene gas industry has attracted attention as a potential alternative calcium source for calcium nitrate fertilizer production. The reutilization of this waste contributes to waste valorization and environmental sustainability while reducing industrial waste disposal. The high calcium content of  $\text{Ca(OH)}_2$  waste enhances its suitability as a feedstock for calcium nitrate synthesis. Therefore, X-Ray Fluorescence (XRF) characterization was conducted at the Advanced Mineral and Materials Laboratory, Universitas Negeri Malang, to determine its composition.

**Table 3.** Elemental Composition of  $\text{Ca(OH)}_2$  Waste Determined by X-Ray Fluorescence (XRF) Analysis

| No | Component | Content (%w/w) |
|----|-----------|----------------|
| 1  | S         | 0.55           |
| 2  | Ca        | 98.71          |
| 3  | Fe        | 0.38           |
| 4  | Co        | 0.086          |
| 5  | Cu        | 0.03           |
| 6  | Er        | 0.1            |
| 7  | Lu        | 0.13           |

Calcium (Ca) was confirmed as the principal element in the sample by XRF analysis, with a measured abundance of 98.71%. The predominance of calcium indicates that the  $\text{Ca(OH)}_2$  waste possessed substantial purity and could serve effectively as a  $\text{Ca}^{2+}$  ion source for calcium nitrate fertilizer production. Other elements were present only in smaller amounts, including sulfur (S) 0.55%, iron (Fe) 0.38%, cobalt (Co) 0.086%, copper (Cu) 0.03%, erbium (Er) 0.10%, and lutetium (Lu) 0.13%. These

elements indicate the presence of minor impurities, which may have originated from the raw material, acetylene production process, or sample handling.

#### 3.2. Spectrophotometric and Inductively Coupled Plasma (ICP) Analysis of Calcium Nitrate Fertilizer

Nutrient content analysis of calcium nitrate fertilizer was conducted at the Environmental Quality Management Laboratory, Department of Environmental Engineering, Faculty of Civil, Planning, and Geo Engineering (FTSPK), ITS to determine the nitrogen (N) and calcium (Ca) contents in the product. The nitrogen content was analyzed using the spectrophotometric method, while the calcium content was determined using Inductively Coupled Plasma (ICP) analysis.

**Table 4.** Nitrogen (N) and Calcium (Ca) Content of Calcium Nitrate Fertilizer

| Temperature (°C) | $\text{HNO}_3$ Concentration (N) | Nitrogen Content (%) | Calcium Content (%) |
|------------------|----------------------------------|----------------------|---------------------|
| 50               | 1                                | 10.61                | 17.01               |
|                  | 1.25                             | 10.9                 | 18.85               |
|                  | 1.5                              | 11.25                | 20.22               |
|                  | 1.75                             | 11.67                | 21.11               |
|                  | 2                                | 12.11                | 23.6                |
| 60               | 1                                | 12.72                | 18.41               |
|                  | 1.25                             | 12.88                | 19.15               |
|                  | 1.5                              | 13.37                | 20.65               |
|                  | 1.75                             | 13.6                 | 21.42               |
|                  | 2                                | 14.54                | 24.85               |
| 70               | 1                                | 15.25                | 19.05               |
|                  | 1.25                             | 15.81                | 19.89               |
|                  | 1.5                              | 16.74                | 21.91               |
|                  | 1.75                             | 17.8                 | 22.31               |
|                  | 2                                | 18.26                | 25.31               |
| 80               | 1                                | 18.75                | 19.94               |
|                  | 1.25                             | 19                   | 20.58               |
|                  | 1.5                              | 20.47                | 22.11               |
|                  | 1.75                             | 21.28                | 23.43               |
|                  | 2                                | 21.77                | 26.07               |
| 90               | 1                                | 22.05                | 21.02               |
|                  | 1.25                             | 22.28                | 21.31               |
|                  | 1.5                              | 22.46                | 22.5                |
|                  | 1.75                             | 22.92                | 24.41               |
|                  | 2                                | 23.47                | 26.39               |

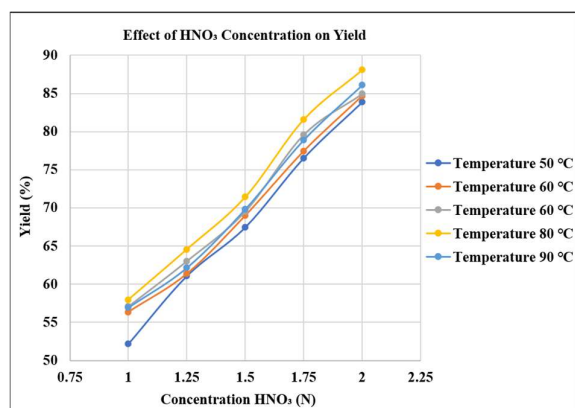
Based on Table 4, it can be observed that the best results in the production of solid calcium nitrate fertilizer from  $\text{Ca(OH)}_2$  waste using the crystallization method were obtained at a reaction temperature of 70 °C and  $\text{HNO}_3$  concentration of 1.5 N, yielding a calcium content of 21.91% and a nitrogen content of 16.74%. The produced calcium nitrate fertilizer meets the SNI 02-2806-

1992 standard, which specifies a calcium content of 19–22% and a minimum nitrogen content of 15.5 %, and is completely soluble in water [12].

### 3.3. Effect of Nitric Acid Concentration on Calcium Nitrate Yield

**Table 5.** Calcium Nitrate Yield (%) Obtained at Different Nitric Acid (HNO<sub>3</sub>) Concentrations and Reaction Temperatures.

| HNO <sub>3</sub> Concentration (N) | Temperature (°C) | Theoretical Weight (gr) | Actual Weight (gr) | Yield (%) |
|------------------------------------|------------------|-------------------------|--------------------|-----------|
| 1                                  | 50               | 40                      | 20.8723            | 52.18075  |
|                                    | 60               |                         | 22.5498            | 56.3745   |
|                                    | 70               |                         | 22.8376            | 57.094    |
|                                    | 80               |                         | 23.1941            | 57.98525  |
|                                    | 90               |                         | 22.766             | 56.915    |
| 1.25                               | 50               | 40                      | 24.4301            | 61.07525  |
|                                    | 60               |                         | 24.5499            | 61.37475  |
|                                    | 70               |                         | 25.214             | 63.035    |
|                                    | 80               |                         | 25.8333            | 64.58325  |
|                                    | 90               |                         | 24.8544            | 62.136    |
| 1.5                                | 50               | 40                      | 26.9874            | 67.4685   |
|                                    | 60               |                         | 27.6042            | 69.0105   |
|                                    | 70               |                         | 27.8256            | 69.564    |
|                                    | 80               |                         | 28.5775            | 71.44375  |
|                                    | 90               |                         | 27.9278            | 69.8195   |
| 2                                  | 50               | 40                      | 30.6154            | 76.5385   |
|                                    | 60               |                         | 30.9835            | 77.45875  |
|                                    | 70               |                         | 31.8402            | 79.6005   |
|                                    | 80               |                         | 32.6416            | 81.604    |
|                                    | 90               |                         | 31.5595            | 78.89875  |
| 2.5                                | 50               | 40                      | 33.5455            | 83.86375  |
|                                    | 60               |                         | 33.8751            | 84.68775  |
|                                    | 70               |                         | 33.9794            | 84.9485   |
|                                    | 80               |                         | 35.2381            | 88.09525  |
|                                    | 90               |                         | 34.4379            | 86.09475  |



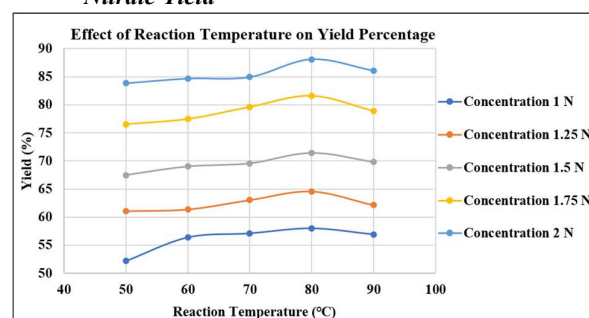
**Figure 2.** Effect of Nitric Acid (HNO<sub>3</sub>) Concentration on Yield of Calcium Nitrate Fertilizer

Based on Figure 2, an increase in nitric acid (HNO<sub>3</sub>) concentration resulted in an increase in the calcium nitrate yield at all reaction temperatures investigated. A positive correlation was observed between HNO<sub>3</sub> concentration and calcium nitrate yield, indicating that higher HNO<sub>3</sub> concentrations were associated with greater product yields. At a reaction temperature of 50°C, increasing the HNO<sub>3</sub> concentration from 1 N to 2 N gradually increased the calcium nitrate yield from 52.18% to 83.86%. A similar trend was also observed at the other reaction temperatures investigated. At 60°C, the calcium nitrate yield increased from 54.50% at an HNO<sub>3</sub> concentration of 1 N to 84.69% at 2 N. Similarly, at 70°C, the yield increased from 57.09% to 84.95%. Meanwhile, the highest yield was obtained at 80°C, reaching 88.10% at an HNO<sub>3</sub> concentration of 2 N. At 90°C, although a slight decrease in yield was observed compared to 80°C, the effect of increasing HNO<sub>3</sub> concentration remained evident, where the yield increased from 56.92% to 86.09% as the acid concentration increased from 1 N to 2 N.

The observed increase in yield suggests that nitric acid concentration plays a critical role in promoting calcium nitrate formation through enhanced reaction conversion. According to Karshyga (2022), increasing the concentration of nitric acid in the reaction medium enhances the reaction between calcium hydroxide contained in the waste material and nitric acid, resulting in a greater formation of calcium nitrate. At low acid concentrations, the availability of H<sup>+</sup> ions is insufficient to react completely with the calcium hydroxide present, thereby limiting the extent of the reaction. In contrast, increasing the HNO<sub>3</sub> concentration increases the availability of H<sup>+</sup> and NO<sub>3</sub><sup>-</sup> ions in the solution, promoting a more extensive formation of calcium nitrate [20].

From a theoretical perspective, raising the concentration of reactants improves the likelihood of effective molecular collisions and subsequently increases reaction kinetics [21]. Furthermore, a higher concentration gradient between the acid solution and the waste particle surface provides a greater driving force for mass transfer. This condition accelerates the dissolution of calcium-containing compounds from the waste material and facilitates the formation of Ca<sup>2+</sup> ions, which subsequently react with nitrate ions to form calcium nitrate [22]. Therefore, using higher concentrations of HNO<sub>3</sub> increases the likelihood of forming a larger actual crystal mass, resulting in a higher percentage yield.

### 3.4. Effect of Reaction Temperature on Calcium Nitrate Yield



**Figure 3.** Effect of Reaction Temperature on Calcium Nitrate of Yield Calcium Nitrate Fertilizer

Based on (Figure 3), the effect of reaction temperature on calcium nitrate yield shows a different trend compared to the effect of  $\text{HNO}_3$  concentration. While increasing  $\text{HNO}_3$  concentration consistently increases yield, temperature variation exhibits both increasing and decreasing trends under different conditions. The yield increases up to a certain temperature, then decreases at higher temperatures. At a concentration of 1 N, an increase in temperature from 50°C to 80°C led to an increase in yield from 52.18% to 57.99%, followed by a decrease to 56.92% at 90°C. A similar trend was observed at 1.25 N, where the yield increased from 61.08% at 50°C to 64.58% at 80°C, then decreased to 62.14% at 90°C. The same pattern was observed at 1.5 N and 1.75 N, where the percentage yield increased with temperature up to 80°C, then decreased at higher temperatures. At a concentration of 2 N, increasing the temperature up to 80°C resulted in the highest yield of 88.10%. However, when the temperature was further increased to 90°C, the yield decreased to 86.09%.

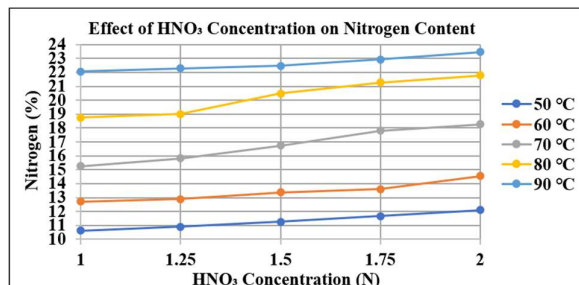
The increase in percentage yield within the temperature range of 50–80°C indicates that the addition of thermal energy positively influences the calcium nitrate formation reaction. According to Fogler (2016), an increase in temperature raises the kinetic energy of molecules, thereby increasing both the frequency and energy of collisions between reactants. This condition enhances the reaction rate constant, resulting in a faster conversion of reactants into products. At higher temperatures, the solution's viscosity tends to decrease, thereby improving mass transfer between the acid solution and the surfaces of waste particles. The reduction in viscosity facilitates the diffusion of  $\text{H}^+$  ions toward the solid surface and accelerates the release of  $\text{Ca}^{2+}$  ions into the solution. As a result, the dissolution rate of the waste material and the formation of calcium nitrate increase, leading to a higher product yield [21].

However, increasing temperature does not always have a positive effect. The experimental results show that after reaching approximately 80°C, the yield decreases at 90°C. This phenomenon indicates the existence of an optimal operating temperature for calcium nitrate production. The reduction in yield at higher temperatures is primarily due to the volatility of nitric acid. According to Treybal (1980), operating temperatures near the solution's boiling point may cause partial evaporation of  $\text{HNO}_3$  before it fully reacts with the solid waste. This evaporation reduces the effective acid concentration in the system, thereby decreasing the availability of  $\text{H}^+$  ions for the reaction. As a result, the conversion of calcium hydroxide is not completed optimally, leading to a lower amount of product formed compared to the optimum temperature condition [22].

Based on Figure 4, increasing nitric acid ( $\text{HNO}_3$ ) concentration from 1 N to 2 N consistently enhances the nitrogen content in calcium nitrate fertilizer across all reaction temperatures, indicating that acid concentration plays a significant role in improving reaction performance. At 50°C, nitrogen content increased from 10.61% to 12.11% with increasing  $\text{HNO}_3$  concentration. A similar trend was observed at 60°C, with the nitrogen content increasing from 12.72% to 14.54%. At 70°C, it increased from 15.25% to 18.26%, while at 80°C it increased from 18.75% to 21.77%. The highest nitrogen content was obtained at 90°C, increasing from 22.05% to 23.47%. This behavior can be explained by the increased availability of nitrate ions ( $\text{NO}_3^-$ ), which enhances the reaction kinetics between calcium sources and nitric acid. Higher acid concentration

increases the driving force for mass transfer and promotes more frequent effective collisions between reactant species, thereby accelerating the formation of calcium nitrate ( $\text{Ca}(\text{NO}_3)_2$ ) in the solution phase.

### 3.4. Effect of Nitric Acid ( $\text{HNO}_3$ ) Concentration on Nitrogen Content



**Figure 4.** Nitric Acid Concentration Influence on Nitrogen Content in Calcium Nitrate Fertilizer

The increase in nitrogen levels along with increasing  $\text{HNO}_3$  concentration is in accordance with theory, because the number of nitrate ions available for the formation of calcium nitrate ( $\text{Ca}(\text{NO}_3)_2$ ) is greater. These results are in line with the findings of Musa (2024) who stated that fertilizer nitrogen levels are influenced by reaction conditions and the amount of reactants used [23]. In addition, the use of 25%  $\text{NH}_4\text{OH}$  in the neutralization stage also has the potential to offer nitrogen through ammonium ions ( $\text{NH}_4^+$ ) and the possibility of  $\text{NH}_4\text{NO}_3$  formation. Therefore, the values obtained in this study represent the total nitrogen content [24].

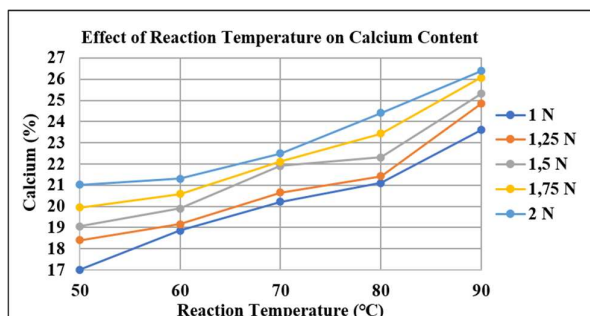
The results showed that several experimental conditions met the SNI 02-2806-1992 specification, which sets the minimum nitrogen content at 15.5%. Nitrogen content is an important quality parameter in calcium nitrate fertilizer because it is present in the form of nitrate, which is readily available for plant uptake [12]. This requirement was met at a reaction temperature of 70°C for  $\text{HNO}_3$  concentrations (1.25 N, 1.5 N, 1.75 N, and 2 N), and at 80°C and 90°C at all variations of  $\text{HNO}_3$  concentrations (1 N, 1.25 N, 1.5 N, 1.75 N, and 2 N).

In contrast, at lower reaction temperatures of 50°C and 60°C, nitrogen content values were generally lower and did not consistently meet the minimum SNI requirements, particularly at lower  $\text{HNO}_3$  concentrations. However, an upward trend was still observed with increasing acid concentration, indicating that greater availability of nitrate ions increases nitrogen incorporation into the final product, even at lower temperatures. Overall, the results indicate that nitrogen content is strongly influenced by both reaction temperature and acid concentration, where higher operating conditions promote more effective formation of nitrate-based nitrogen. The highest nitrogen content obtained in this study was 23.47%.

### 3.5. Effect of Reaction Temperature on Calcium (Ca) Content

Based on the (Figure 5), reaction temperature significantly influences the calcium (Ca) content in calcium nitrate fertilizer. At a concentration of 1 N, increasing the temperature from 50°C

to 90°C increased the calcium content from 17.01% to 21.02%. A similar trend was observed at 1.25 N, where the calcium content increased from 18.85% at 50°C to 21.31% at 90°C. At 1.5 N, the calcium content increased from 20.22% to 22.50%, while at 1.75 N it increased from 21.11% to 24.41%. The highest increase was observed at 2 N, where the calcium content rose from 23.60% at 50°C to 26.39% at 90°C. These results indicate that higher reaction temperatures tend to increase the calcium content in the produced calcium nitrate fertilizer.



**Figure 5.** Reaction Temperature Influence on Calcium (Ca) Content in Calcium Nitrate Fertilizer

This behavior can be explained by the increase in molecular kinetic energy at higher temperatures, which enhances collision frequency and collision effectiveness between reactant species. According to chemical reaction engineering principles, higher temperature increases reaction rate constants and accelerates reaction rates in heterogeneous systems [21, 25]. As a result, the reaction between calcium-containing compounds in the waste and nitric acid is accelerated, leading to higher dissolution rates and increased formation of  $\text{Ca}^{2+}$  ions in the liquid phase.

In addition to reaction kinetics, temperature also improves mass transfer between the solid surface and the acid solution. Higher temperatures reduce diffusion resistance in the liquid boundary layer and enhance the transport rate of reactants toward the solid surface. This phenomenon is consistent with the shrinking core model for solid-liquid reactions, where both surface reaction and diffusion resistance govern the overall reaction rate [26]. These combined effects increase  $\text{Ca}^{2+}$  availability in the system, thereby increasing calcium incorporation into calcium nitrate products.

From a process perspective, the observed increase in calcium content indicates an improved overall reaction extent at elevated temperatures, where both kinetic and mass-transfer limitations are reduced, resulting in more efficient conversion of calcium-based raw materials.

The experimental results show that several operating conditions meet the requirements of SNI 02-2806-1992, which specifies a calcium content range of 19–22% [12]. The experimental results show that several operating conditions meet the requirements of SNI 02-2806-1992, which specifies a calcium content range of 19–22% [12]. The operating conditions that satisfy this requirement include 50°C with  $\text{HNO}_3$  concentrations of 1.5 N and 1.75 N, 60°C with 1.25 N, 1.5 N, and 1.75 N, 70°C with 1 N, 1.25 N, and 1.5 N, 80°C with 1 N and 1.25 N, and 90°C with 1 N and 1.25 N.

The operating condition at 70°C and 1.5 N  $\text{HNO}_3$  was selected as the most appropriate because it fully satisfies the SNI calcium content specification while maintaining relatively high calcium and nitrogen values compared to other acceptable

conditions. Although several other conditions yielded higher calcium content, they were not selected because they exceeded the SNI allowable range and therefore did not meet product quality requirements. Under these conditions, the calcium content reached 21.91%, which falls within the specified SNI range. Therefore, the selection is based on compliance with SNI standards and an overall balance of the process rather than on maximum calcium content.

Although ICP and UV-Vis spectrophotometric analysis successfully confirmed the calcium and nitrogen content of synthesized calcium nitrate fertilizer, these methods are insufficient to thoroughly verify the product's formation and structural characteristics. Therefore, additional characterization methods are needed to obtain a more comprehensive material evaluation. Advanced characterization methods such as X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM) are commonly used to complement elemental analysis to confirm successful calcium nitrate synthesis. XRD is used to identify the crystal phase and confirm the formation of calcium nitrate [27, 28]. FTIR spectroscopy is commonly applied to verify the presence of nitrate functional groups, particularly the characteristic  $\text{NO}_3^-$  vibrations in the range of 1380–1450  $\text{cm}^{-1}$  [29]. Meanwhile, SEM is used to observe the morphology and surface structure of crystals, which are important indicators in assessing the success of the crystallization process and product quality [30]. Therefore, further research is recommended to complement characterization using XRD, FTIR, and SEM to obtain more comprehensive validation of the product structure and morphology.

#### 4. CONCLUSION

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) waste generated from the acetylene gas industry has been successfully utilized as an alternative raw material for calcium nitrate fertilizer production through a reaction with nitric acid followed by a crystallization process. The concentration of  $\text{HNO}_3$  and reaction temperature significantly influenced the calcium nitrate yield, calcium content, and nitrogen content of the obtained product. The best condition obtained in this study was achieved at a reaction temperature of 70 °C with an  $\text{HNO}_3$  concentration of 1.5 N, yielding a yield of 69.564%, a calcium content of 21.91%, and a nitrogen content of 16.74%. The resulting calcium nitrate fertilizer meets the requirements of SNI 02-2806-1992, including a calcium content of 19–22% and a minimum nitrogen content of 15.5%. However, further characterization using X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM) is still required to confirm the phases formed. Moreover, the utilization of  $\text{Ca}(\text{OH})_2$  waste supports environmental sustainability by reducing industrial waste while providing potential economic benefits due to the use of low-cost raw materials, although further technical-economic feasibility assessment is recommended.

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