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Synthesis and Characterization of Calcium Phosphate-Based Material Containing P and K Based on Acetylene Gas Waste Raw Material Using Dissolution and Precipitation Methods

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ABSTRACT

Acetylene gas waste possesses considerable potential for reutilization because of its high calcium content, although inadequate management may lead to adverse environmental effects. This research was conducted to synthesize and characterize multinutrient phosphate-based materials derived from acetylene gas waste through dissolution and precipitation techniques. The synthesis involved reacting calcium hydroxide obtained from acetylene gas waste with phosphoric acid (H_3PO_4), followed by a precipitation process using NH_4OH and KOH solutions. The experimental variables included pH values ranging from 7 to 11 and H_3PO_4 concentrations between 15% and 55%. Characterization of the synthesized products was performed using X-Ray Fluorescence (XRF), Scanning Electron Microscopy (SEM), and X-Ray Diffraction (XRD) to evaluate elemental composition, crystal morphology, and crystalline structures. The findings revealed that higher pH values and increased H_3PO_4 concentrations had a significant influence on both the yield and quality of the precipitated products. The maximum yield of 90% was achieved at an H_3PO_4 concentration of 55% and pH 10. SEM observations revealed that the products exhibited a finer, more homogeneous crystal morphology, while XRF analysis confirmed that calcium was the dominant element, accompanied by phosphorus and potassium, indicating the successful synthesis of calcium phosphate-based materials. Furthermore, XRD analysis confirmed the formation of crystalline calcium phosphate compounds. Overall, the study highlights the potential application of acetylene gas waste as an alternative raw material for the production of sustainable and environmentally friendly phosphate fertilizers.

Contribution to Sustainable Development Goals (SDGs):

SDG 9: Industry, Innovation and Infrastructure

SDG 12: Responsible Consumption and Production

SDG 13: Climate Action

1. INTRODUCTION

1.1. Research Background

The continuous growth of industrial operations, particularly in welding and metal fabrication sectors, has led to an increasing amount of acetylene gas waste. This by-product is predominantly composed of calcium hydroxide ($Ca(OH)_2$), making it a promising alternative source of calcium. However, improper disposal of acetylene waste may pose environmental risks.

Therefore, its conversion into phosphate fertilizer raw material offers an effective strategy for waste valorization while supporting sustainable industrial practices [1].

Phosphorus is an essential nutrient required for various physiological processes in plants, including root development, energy transfer, and crop production [2]. In this work, a phosphate-based multinutrient material was synthesized from acetylene gas waste and phosphoric acid (H_3PO_4) through dissolution and precipitation methods. The resulting calcium



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phosphate compounds have potential applications as fertilizer materials with improved nutrient availability [3].

In contrast to earlier studies that primarily investigated phosphate fertilizer production from acetylene waste, this research focuses on the synthesis and characterization of Ca–N–P–K multinutrient materials using acetylene waste as the main calcium source. The effects of phosphoric acid concentration and precipitation pH were systematically evaluated to identify the most favorable synthesis conditions. Furthermore, NH_4OH and KOH were utilized not only as precipitating agents but also as sources of nitrogen and potassium, enabling the production of a more complete multinutrient fertilizer. The influence of pH values ranging from 7 to 11 on product formation and yield was also assessed, providing additional insight into the optimization of the synthesis process [4].

1.2. Literature Review

Acetylene gas waste is an industrial by-product that contains a high concentration of calcium hydroxide ($\text{Ca}(\text{OH})_2$), making it a potential alternative raw material for phosphate fertilizer production. The conversion of this waste into calcium phosphate compounds through dissolution with phosphoric acid offers an environmentally friendly approach for waste utilization and resource recovery [1].

Phosphorus is an essential macronutrient involved in root development, energy transfer, photosynthesis, and crop productivity [2]. Phosphorus deficiency can reduce plant growth and yield; therefore, phosphate fertilizers are widely applied to improve soil fertility. To ensure effectiveness and environmental safety, phosphate fertilizers must meet quality standards regarding nutrient content and physical properties [5].

The synthesis of phosphate-based multinutrient materials involves dissolution of $\text{Ca}(\text{OH})_2$ in H_3PO_4 , where the reactant ratio significantly affects phosphate formation and nutrient availability [3]. Process variables such as temperature, stirring speed, and reaction time also influence crystal growth and product characteristics [6].

Precipitation is a simple and cost-effective method for producing uniform particles. In this study, NH_4OH and KOH were used as precipitating agents to regulate pH and promote phosphate crystallization [7]. Precipitation efficiency and product formation are strongly influenced by pH during the process [8].

The synthesized materials were characterized using X-Ray Fluorescence (XRF) to determine elemental composition [9], Scanning Electron Microscopy (SEM) to examine surface morphology and particle characteristics [10], and X-Ray Diffraction (XRD) to identify crystalline phases and structural properties [11]. These analytical techniques provide essential information for evaluating the quality and potential application of the synthesized phosphate materials as fertilizers.

1.2.1. Dissolution Method

The dissolution method applied in this study represented the initial stage in the synthesis of multinutrient phosphate-based materials derived from acetylene gas waste. This process involved dissolving acetylene gas waste, which contains calcium hydroxide ($\text{Ca}(\text{OH})_2$), using phosphoric acid (H_3PO_4). The main purpose of this method was to transform calcium compounds contained in the waste into calcium phosphate compounds that could potentially be utilized as phosphate fertilizer materials [1].

Prior to the dissolution process, the acetylene gas waste underwent several preparation steps, including drying, grinding, and sieving to a particle size of 100 mesh to obtain more uniform particles and improve reaction efficiency. After preparation, 30 grams of acetylene gas waste were reacted with 200 mL of phosphoric acid solution at concentrations of 15%, 25%, 35%, 45%, and 55%. The dissolution process was carried out using a magnetic stirrer at 100°C for 1 hour with a stirring speed of 200 rpm.

During the dissolution stage, calcium hydroxide reacted with phosphoric acid to form calcium phosphate compounds and water through a heterogeneous reaction involving both solid and liquid phases:



In this reaction, phosphoric acid acted as both a solvent and a phosphate source, supplying phosphate ions that reacted with calcium ions to form calcium phosphate compounds. The ratio of $\text{Ca}(\text{OH})_2$ to H_3PO_4 strongly affects the solubility and types of phosphate compounds formed [3]. An appropriate reactant composition can produce mono-calcium phosphate with higher solubility and improved phosphorus availability for plants.

Several operational factors also influenced the dissolution process, including temperature, stirring speed, and dissolution time. Increasing temperature enhances the solubility of most solid compounds and accelerates reaction rates. Meanwhile, higher stirring speeds increase molecular collisions, leading to smaller crystal sizes, whereas longer stirring times promote crystal growth and improve product formation [8].

Following the dissolution stage, the resulting mixture was filtered to separate the filtrate from the residue, then the precipitation stage was performed using NH_4OH and KOH solutions. Therefore, the dissolution process played a crucial role in determining the availability of calcium and phosphate ions required for the formation of multinutrient Ca–N–P–K materials in the subsequent synthesis stage [9].

1.2.2. Precipitation Method

The precipitation method used in this study was carried out after the dissolution stage to synthesize multinutrient phosphate-based materials from acetylene gas waste. Precipitation is a process in which dissolved substances react with precipitating agents to form solid particles or precipitates. According to [6], the precipitation method is widely used because it is relatively simple, economical, easy to control, and capable of producing fine particles with uniform morphology and even nano-sized structures.

In this research, the filtrate obtained from the dissolution process was precipitated with 1 N ammonium hydroxide (NH_4OH) and 1 N potassium hydroxide (KOH). The addition of NH_4OH and KOH was performed gradually until the desired pH values of 7, 8, 9, 10, and 11 were achieved. NH_4OH and KOH were selected because they are alkaline compounds that are easily soluble in water and effective in controlling the pH of the solution during crystal formation. The increase in pH during precipitation significantly influenced crystal formation, calcium oxide content, and the amount of precipitate produced. Higher pH values can enhance calcium oxide formation and improve precipitation efficiency.

During precipitation, calcium phosphate compounds formed by dissolution reacted with NH_4OH and KOH to produce multinutrient phosphate compounds such as CaNH_4PO_4 and CaKPO_4 . The reactions occurring during the precipitation stage are shown below:



The precipitation process was also influenced by several operational parameters, including pH, stirring speed, and stirring time. Higher stirring speed increases molecular collisions and nucleation rates, resulting in smaller crystal particles and improved homogeneity of the synthesized product. In addition, longer stirring time allows more interactions among reactant particles, leading to greater product formation and larger crystal growth [10].

After precipitation, the resulting precipitate was separated from the filtrate through filtration and subsequently dried in an oven at 110°C for 3 hours before further characterization. This precipitation stage played an essential role in determining the yield, morphology, and crystalline structure of the synthesized multinutrient phosphate materials [4].

1.3. Research Objective

This study was conducted to synthesize and characterize multinutrient phosphate-based materials utilizing acetylene gas waste through dissolution and precipitation processes. The research also focused on examining the influence of different H_3PO_4 concentrations and precipitation pH values on the yield, composition, and properties of the synthesized products. Furthermore, this study aimed to analyze the elemental composition, crystal morphology, and crystalline structure of the obtained materials using XRF, SEM, and XRD characterization methods to evaluate their potential as sustainable and environmentally friendly phosphate fertilizer materials.

2. MATERIALS AND METHODS

In this research, acetylene gas waste containing calcium hydroxide ($\text{Ca}(\text{OH})_2$) was employed as the main raw material for synthesizing multinutrient phosphate-based compounds. Additional chemicals used in the study were phosphoric acid (H_3PO_4), ammonium hydroxide (NH_4OH), and potassium hydroxide (KOH). Before the synthesis stage, the acetylene gas waste underwent a preparation process consisting of drying at 110°C to minimize moisture content, followed by grinding and sieving through a 100-mesh sieve to obtain more homogeneous particle sizes. Afterwards, 30 g of the prepared material was mixed with 200 mL of phosphoric acid solution at concentrations of 15%, 25%, 35%, 45%, and 55%. The dissolution process was performed at 100°C for 1 hour using a magnetic stirrer operating at 200 rpm. During this stage, calcium hydroxide reacted with phosphoric acid to produce calcium phosphate compounds. The resulting mixture was then filtered to separate the filtrate and residue. Subsequently, the filtrate was subjected to a precipitation process by adding 1 N NH_4OH and 1 N KOH solutions until the pH reached 7, 8, 9, 10, and 11.

The acetylene gas waste was collected from a local welding workshop in Surabaya, Indonesia. Prior to use, the waste was oven-dried at 110°C to remove free moisture. Drying was carried out under atmospheric air conditions. The suspension obtained after dissolution and precipitation was separated using vacuum filtration with filter paper. The precipitate was then dried in an oven at 110°C for 3 h before characterization. The elemental composition of the synthesized products was analyzed using an X-Ray Fluorescence (XRF) spectrometer (Rigaku NEX CG, Japan). Surface morphology was observed using a Scanning Electron Microscope (SEM) (JEOL JSM-6510LA, Japan). The crystalline phases were identified using X-Ray Diffraction (XRD) (Rigaku MiniFlex 600, Japan) with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 15 mA. Diffraction patterns were recorded over a 2θ range of $5\text{--}60^\circ$ with a scanning rate of 2° min^{-1} and a step size of 0.02° .

3. RESULT AND DISCUSSION

3.1 Characterization of Acetylene Gas Waste as Raw Material (XRF)

Before the synthesis process was carried out, preliminary characterization using X-Ray Fluorescence (XRF) was conducted to determine the chemical composition, as well as the dominant and minor elements present in the acetylene gas waste raw material.

Table 1. XRF analysis of acetylene gas waste using Rigaku NEX CG X-Ray Fluorescence spectrometer

No	Jenis Uji (Oksida)	Komposisi (%)	Jenis Uji (Unsur)	Komposisi (%)
1	SO_3	2.05	S	0.995
2	K_2O	0.23	K	0.255
3	CaO	95.55	Ca	96.35
4	TiO_2	0.175	Ti	0.165
5	MnO	0.035	Mn	0.04
6	Fe_2O_3	1.6	Fe	1.765
7	CuO	0.085	Cu	0.1
8	SrO	0.13	Sr	0.17
9	BaO	0.1	Ba	0.135
Total		99.955		99.975

Based on the XRF analysis results, the acetylene gas waste is highly dominated by Calcium Oxide (CaO) at 95.55% and a calcium (Ca) element concentration of 96.35%. This dominant CaO fraction indicates that the acetylene gas waste is chemically dominated by calcium compounds, which aligns with the expected primary characteristic $\text{Ca}(\text{OH})_2$ after undergoing thermal dehydration. This high fraction highlights the massive potential of the raw material to serve as a calcium source for the dissolution reaction with H_3PO_4 in forming phosphate-based multinutrient materials. In addition to the main component, the analysis identified several minor elements (impurities) such as SO_3 (2.05%), Fe_2O_3 (1.60%), along with trace concentrations of TiO_2 , MnO , CuO , SrO , and BaO . Although present in small concentrations, these minor elements require careful consideration as they may affect dissolution reactivity, precipitate

phase formation, and the chemical properties of the final product—particularly regarding phosphate binding and material stability [12].

3.2 Effect of H_3PO_4 Concentration and pH on Product Yield

The yield of Ca-N-P-K was determined by comparing the weight of the obtained multinutrient precipitate against the initial weight of the acetylene gas waste (which was fixed at 30 grams).

The product yield was calculated using Equation :

$$\text{Yield (\%)} = \frac{m_{\text{product}}}{m_{\text{raw material}}} \times 100 \quad (4)$$

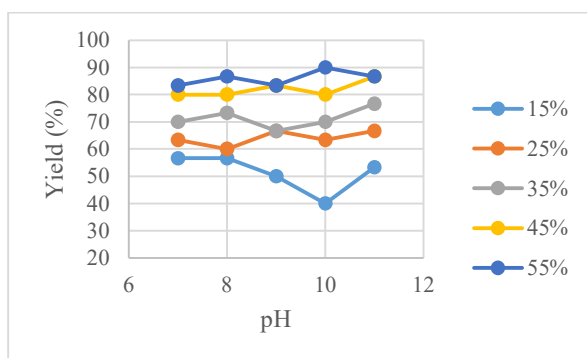


Fig. 1. Effect of H_3PO_4 Concentration and pH on Product Yield

The experimental data indicates that the precipitation yield increases significantly with higher concentrations of H_3PO_4 . The highest yield of 90% was achieved using a 55% H_3PO_4 concentration at pH 10, whereas the lowest yield of 40% occurred at a low concentration of 15%. This trend demonstrates that higher acid concentrations supply more phosphate ions to react with calcium, thereby increasing the formation of calcium-phosphate precipitates [13]. Moreover, the pH variations reveal that moderately alkaline conditions (pH 9–11) support the formation of more abundant and stable precipitates, corresponding to pH-driven changes in phosphate speciation within the solution [14].

In a calcium-phosphate precipitation system, precipitate formation is directly driven by the concentrations of phosphate ions PO_4^{3-} , calcium ions Ca^{2+} , and pH levels. Higher concentrations of phosphate ions increase the solid formation reaction because more ions are available to interact with Ca^{2+} to build a less soluble solid phase, particularly under high concentrations and moderate-to-alkaline pH >8 [24]. Furthermore, studies show that while initial phases show adsorption dominance at lower pH and low phosphate concentrations, higher pH levels markedly improve calcium-phosphate precipitation by reducing inhibitory effects from interfering ions like carbonate at pH >9 [16]. These findings are consistent with prior research utilizing acetylene gas waste for Calcium-Ammonium-Phosphate (CAP) production, which concluded that pH 10 provided the best nutrient characteristics and higher yields, confirming that pH and H_3PO_4 concentration optimization are key parameters for effective multinutrient precipitation from industrial waste [17].

3.3 Morphological Characteristics of Synthesized Product (SEM)

The observation confirms the successful formation of calcium phosphate particles with relatively homogeneous morphology. However, the SEM analysis was intended to illustrate the morphology of the synthesized material and should not be interpreted as representing the optimum synthesis condition determined from the yield experiment. The crystal morphology formed under each treatment showed clear distinctions that closely correlated with the resulting yield percentage :

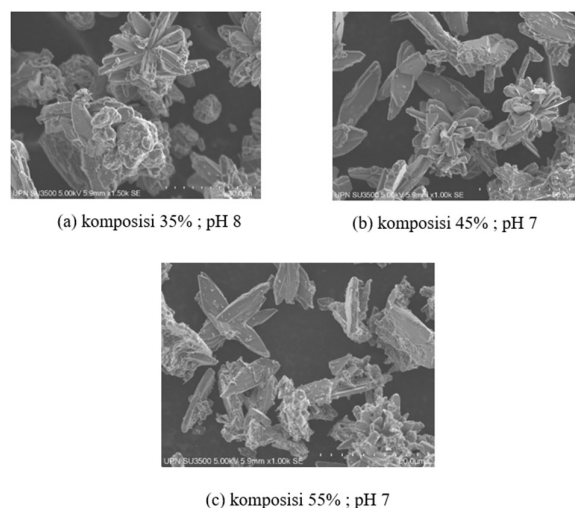


Fig. 2. SEM micrographs of synthesized calcium phosphate products at different synthesis conditions (magnification 5000×, scale bar = 10 μm)

The chemical precipitation process relies heavily on the solution's pH to control how fast the raw calcium and phosphate ions break apart and re-form into stable multinutrient compounds. At a 35% yield (conducted at pH 8), the system is in an unstable state because a neutral-to-low alkaline environment does not provide enough thermodynamic energy to create a sufficient amount of new crystal nuclei. This lack of crystal "seeds" causes the limited reactive ions to heavily crowd and continuously deposit onto a few select spots, resulting in large, rough, and irregular clumped masses (agglomerates) [18]. At a 45% yield (conducted at pH 7), the chemical equilibrium shifts. At this point, the stable neutral pH environment slows the ionic growth rate and balances it against the rising number of new nucleation seeds. With more nucleation sites available throughout the solution, the massive raw clumps naturally fracture into smaller, more uniform particles with noticeably smoother surfaces [19]. Finally, the process reaches its optimal 55% yield (also observed at pH 7), where the combination of an optimal acid concentration and a balanced neutral pH triggers a highly efficient precipitation mechanism. Under these optimal conditions, millions of tiny crystal nuclei snap into existence simultaneously throughout the entire matrix, effectively dispersing the available nutrient ions so evenly that this completely halts uncontrolled clumping [20]. This yields an extremely homogenous, stable, and highly organized microstructure made up of perfectly uniform fine particles [21].

Overall, these changes prove that crystal quality and structural order improve alongside precipitation efficiency.

Literature supports this [22], indicating that while low precipitation numbers cause crystals to grow into irregular aggregates, optimized conditions (such as high pH and adequate concentration) accelerate the transition from the dissolution phase into stable crystals, encouraging fast nucleation that yields finer, highly homogenous, and evenly distributed stable particles [21].

3.4 Chemical Composition Analysis of Product (XRF)

The XRF analysis was carried out on the product synthesized at 45% H_3PO_4 and pH 7. Therefore, the elemental composition reported in this section represents only the selected characterization sample and should not be interpreted as the elemental composition of the optimum-yield product.

Table2 XRF analysis of synthesized calcium phosphate product using Rigaku NEX CG X-Ray Fluorescence spectrometer

Compound	Concentration
P_2O_5	45.1%
K_2O	0.12%
CaO	48.2%

Additionally, P_2O_5 was found in a substantial amount at 45.1%. Although slightly lower than CaO , its significant presence confirms the successful reaction between Ca^{2+} and PO_4^{3-} ions to yield calcium phosphate. The resulting high Ca/P ratio indicates a tendency to form a more basic, calcium-rich calcium phosphate phase, which typically occurs under alkaline precipitation conditions. Meanwhile, K_2O was detected at a minor concentration of 0.12%. Though small, the inclusion of K^+ ions enhances the final product's nutritional value, aligning with the goal of producing a multinutrient fertilizer base. The dominance of calcium over phosphorus and potassium occurs because Ca^{2+} ions precipitate much faster under alkaline conditions into stable solids, while portions of phosphate and potassium remain dissolved in the solution phase if saturation has not been fully reached [16].

When compared to the national standard for solid NPK fertilizer (SNI-2803-2010), the precipitate does not yet fully meet commercial criteria. The SNI standard mandates the presence of Nitrogen (N), Phosphorus (P_2O_5), and Potassium (K_2O), with a minimum combined total of 30%. The current precipitate contains no Nitrogen, and the individual P and K contents are well below the minimum commercial thresholds [26]. Nevertheless, the product successfully serves its main research objective: creating a phosphate-based multinutrient fertilizer base [27]. The high Ca content coupled with P and trace K provides a valuable chemical structure to act as a soil nutrient source, making it highly viable for further development as an alternative fertilizer formulation blend [23].

3.5 Crystalline Phase Identification (XRD ICDD/JCPDS)

Phase crystalline characterization via XRD peak-matching with ICDD/JCPDS standards confirmed the formation of a high-quality calcium phosphate multinutrient material derived from the reaction between acetylene waste CaO and H_3PO_4 .

Table. 3. XRD diffraction pattern of synthesized calcium phosphate recorded using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$), scanned over $2\theta = 5\text{--}60^\circ$

No	2θ ($^\circ$)	Height (cps)	Nama Pupuk	Fase & Unsur ICDD/JCPDS	XRF Terkait	Kontribusi Komposisi	Kecocokan (%)
1	8.09, 10.92	2874	DCP (TSP)	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ (09-0077)	Ca (96.35%)	Fase utama dari CaO	95
2	15.33, 5.77	3186	DCP (TSP)	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ (09-0077)	Ca (96.35%)	Matriks Ca -fosfat	98
3	17.39, 5.09	1958	Monetite	CaHPO_4 (70-0360)	Ca (96.35%)	Fase anhidrat	92
4	23.33, 3.81	5753	DCP (TSP)	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ (09-0077)	Ca (96.35%)	Peak dominan CaO	97
5	33.54, 2.67	4242	Monetite	CaHPO_4 (70-0360)	Ca (96.35%)	Dehidratasi parsial	94

XRD analysis verified the successful formation of calcium phosphate phases in the synthesized fertilizer material. The diffraction pattern revealed characteristic reflections attributable to Dicalcium Phosphate Dihydrate (DCPD, JCPDS 09-0077) at 2θ values of 8.09° , 15.33° , and 23.33° , with the strongest diffraction intensity observed at 23.33° . These peaks indicate that DCPD is one of the principal crystalline phases present in the product. In addition, diffraction peaks detected at 17.39° and 33.54° corresponded to Monetite (JCPDS 70-0360), confirming the presence of anhydrous calcium phosphate. The coexistence of DCPD and Monetite suggests that calcium originating from acetylene gas waste reacted effectively with phosphoric acid during the synthesis process [25]. The formation of Monetite may also be associated with partial dehydration of DCPD during synthesis or sample handling. Phase identification was carried out by comparing the diffraction peaks with standard reference patterns. However, because quantitative phase analysis was not performed, the exact phase composition and purity of the product cannot be determined solely from the XRD data.

4. CONCLUSION

Overall, this study demonstrates the successful synthesis of calcium phosphate-based materials from acetylene gas waste via dissolution and precipitation. Although the synthesized material shows potential as a phosphate fertilizer precursor, further investigations, including nutrient availability, nutrient-release behavior, and plant growth evaluations, are required before its practical application as a fertilizer. One limitation of this study is that the experiments were conducted without replicate measurements; therefore, statistical analyses, including standard deviation and significance testing, were not performed. Future work will include replicated experiments to improve the statistical reliability of the results.

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