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The Synthesis and Characterization of A New Composite Material Ca-Mg-NH_4-PO_4 Dolomite-Based for Effective Multinutrient Fertilizer in Plant Growth

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Abstract

The rapid expansion of Indonesia's agricultural sector has led to a surge in fertilizer demand. Dolomite lime shows potential as a promising alternative material to enhance fertilizer quality. This study aims to synthesize and characterize a Ca-Mg-NH_4-PO_4 composite using dolomite as a precursor through precipitation. The synthesis was successfully achieved through several stages, beginning with dolomite preparation, dissolution in 5 N phosphoric acid, and subsequent precipitation with ammonium hydroxide at pH 8, 30°C, with a stirring speed of 450 rpm. The resulting composite was analyzed using various analytical techniques to evaluate its structural, morphological, and elemental compositions. X-ray diffraction (XRD) results indicated the formation of struvite crystals at positions $2\theta = 20.828°$, 30.539°, 33.234°, brushite crystals at position $2\theta = 11.650°$, and impurity compounds that appeared during the synthesis process. Impurities affecting crystal purity were detected but could be minimized through washing. Scanning Electron Microscopy (SEM) analysis revealed a monoclinic crystal morphology with particle sizes ranging from 4.32 µm to 579 nm. X-ray fluorescence (XRF) and Energy Dispersive X-ray (EDX) analysis verified the presence of Ca, Mg, NH_4, and PO_4 components in the composite. Fourier-Transform Infrared Spectroscopy (FTIR) analysis showed that the composite has P-O-C functional groups at 949 cm^{-1}, 972 cm^{-1}, and 1027 cm^{-1}, and N-H functional groups at 1466 cm^{-1}. Overall, this Ca-Mg-NH_4-PO_4 composite derived from dolomite demonstrates promising characteristics as a multi-nutrient fertilizer, offering a high-quality option to support sustainable agricultural practices.

1. Introduction

Indonesia stands as an agricultural country due to its large agricultural land. According to data from the BPS-Statistic Indonesia, agricultural land in Indonesia reached 7,463,948 hectares in 2019 [1]. The large area of agricultural land has resulted in an increasing fertilizer consumption. According to the Indonesian Fertilizer Manufacturer Association (APPI), fertilizer consumption in Indonesia has ranged from 11 to 12 million tons annually between 2017 and 2022 [2]. To maintain food stability in the agricultural sector, developing and improving the quality of plant fertilizers is necessary. The advancement of fertilizer technology serves as a driving force in exploring alternative products, particularly those that can meet the demand for high-quality materials.

Currently, conventional fertilizers are still widely used in Indonesia's agricultural sector. Conventional fertilizers, primarily composed of nitrogen, phosphorus, and potassium (NPK), have enhanced agricultural productivity. However, their application often leads to environmental issues such as nutrient runoff, soil degradation, and reduced microbial diversity in soils [3, 4]. Studies indicate that while conventional fertilizers can boost immediate crop productivity, they may compromise long-term soil health and sustainability [5]. In contrast, advanced fertilizers are designed to enhance nutrient use efficiency (NUE) and minimize environmental impacts. These innovations allow for more precise nutrient delivery, reducing the risk of over-fertilization [6]. Research indicates that advanced fertilizers can significantly improve NUE compared to conventional options, with studies showing enhanced phosphorus use efficiency (PUE) and reduced ammonia emissions [7, 8].

The weaknesses of conventional fertilizers encourage the need for more environmentally friendly alternatives. Developments in agricultural technology have produced composite fertilizers, such as polyhalite [9]. Composite fertilizers provide multi-nutrients in a single product so that plants get nitrogen, phosphorus, potassium, and other required macronutrients and micronutrients. In addition, the primary advantage of composite fertilizers lies in their nutrients' slow-release nature. This slow-release effect is caused by the strong inter-component bonds within their structure, allowing nutrients to remain steadily available to plants over an extended period, thereby supporting efficient nutrient absorption. This reduces re-fertilization frequency, helps prevent environmental pollution due to fertilizer leaching, and makes composite fertilizers more environmentally friendly and economical in the long run. Its high uptake efficiency and ability to provide diverse nutrients make it a more sustainable option to support Indonesia's agricultural sector [10].

Along with the increasing awareness of the importance of sustainable agricultural practices, research and development of composite fertilizers are gaining more attention. The availability of diverse raw materials and innovations in synthesis methods open opportunities to create more effective and efficient composite fertilizers. Several studies have successfully developed multi-nutrient fertilizers by combining various materials, such as liquid waste from the *Tempe* industry [11], geothermal sludge [12], and waste brine [13]. In addition to raw materials in the form of waste, one of the materials that can be used in the synthesis of multi-nutrient fertilizers is dolomite lime, which has considerable potential and spreads almost evenly throughout the islands of Indonesia.

According to KESDM, dolomite lime is distributed in Aceh, North Sumatra, Banten, Central Java, East Java, North Maluku, East Nusa Tenggara, Central Sulawesi, West Sulawesi, and Southeast Sulawesi [14]. The largest dolomite distribution in East Java, particularly in Gresik, Lamongan, and Tuban [15]. As a natural resource, Dolomite contains CaO, MgO, and several other impurities [16]. In development, dolomite lime is widely used in various industries, especially in agriculture, as a plant fertilizer [17, 18, 19].

Dolomite lime contains calcium nutrients that can benefit plants' growth and development, especially plant roots and shoots. Calcium helps regulate plant permeability, maintain the integrity of the structure and function of plant membranes, regulate ion transpiration, and control ion exchange in plants [20]. Additionally, dolomite contains magnesium, essential for chlorophyll formation. A magnesium deficiency can cause yellowing of leaves, shrinkage, and brittleness [21]. The efficacy of dolomite lime as a dolomite fertilizer in agriculture is currently regarded as relatively limited. This is primarily due to the common practice of supplementing dolomite fertilizer with additional nutrient fertilizers to achieve optimal effectiveness in promoting plant growth [22, 23]. However, excessive nutrient addition can saturate the soil, leading to plant stress [24, 25].

This research tries to overcome the weakness by proposing the development of a Ca-Mg-NH_4-PO_4 composite from dolomite through the precipitation method. Dolomite was chosen as the composite base material due to the availability of considerable dolomite resources in Indonesia. With the addition of ammonium (NH_4) and phosphate (PO_4) elements, the resulting composite is expected to increase dolomite minerals' economic value and quality. Phosphate (PO_4) component plays a crucial role in agricultural systems. Within this component, the nutrient phosphorus (P) acts as a stimulant for root formation, enhances the proliferation of fruit tillers, and facilitates the acceleration of both flowering and fruit ripening processes [26]. The element nitrogen (N) contained in ammonium (NH_4^+) can help accelerate the vegetative growth of plants [27]. A complete combination of nutrients can support plant growth and produce quality products.

In synthesizing multi-nutrient fertilizer composites, the initial crucial step involves selecting an appropriate method. The composite synthesis process consists of two distinct stages: the dolomite dissolution stage and the composite crystal formation stage. The dolomite dissolution stage is undertaken to extract the essential elements of calcium (Ca) and magnesium (Mg) from dolomite. In previous studies, the dissolution method with acid solvents was more widely used [28, 29, 30, 31, 32]. Following the dolomite dissolution stage, the subsequent step involves the formation of composite crystals. Various methods can form composite crystals, including granulation and precipitation. In this study, the precipitation method was chosen due to its ease of operation, relatively low costs, and the ready availability of the precipitant material [33].

Based on this background, this research aims to synthesize and characterize a Ca-Mg-NH_4-PO_4 composite derived from dolomite using various analytical techniques. X-ray diffraction (XRD) analysis was first conducted to assess the crystallinity and identify the mineral phases of the synthesized. Following this, X-ray fluorescence (XRF) analysis was utilized to determine the elemental composition of the composite. Scanning Electron Microscopy (SEM) was employed to examine the surface morphology, while Energy Dispersive X-ray (EDX) analysis provided detailed information about the chemical elements present in the composite. Finally, Fourier-Transform Infrared Spectroscopy (FTIR) was used to identify the functional groups within the composite. This study is expected to make a positive contribution to the development of new materials that have the potential to provide benefits in the agricultural and environmental sectors.

2. Experimental

2.1. Materials and Instrument

The materials used in this research included dolomite lime ($CaMg(CO_3)_2$) obtained from PT Cipta Makmur Pertiwi Gresik, containing MgO (19.75%), CaO (29.5%), SiO_2 (1.75%), Al_2O_3 + Fe_2O_3 (2.08%), and some of the carbonate. Additionally, distilled water (H_2O), phosphoric acid (H_3PO_4, Merck 85%), and ammonium hydroxide (NH_4OH, Merck 25%) were obtained from CV Chemical Indonesia Multi Sentosa, Surabaya. All reagents used were of analytical reagent grade.

The equipment used in this research included a set of standard laboratory glassware (Pyrex), magnetic stirrer (Thermo Scientific), filter paper (Whatman 42), analytical balance (Sartorius BT224S), pH indicator paper (Merck), porcelain mortar (Onemed), and oven. The analytical equipment consisted of XRD (Philips Binary X'Pert MPD), XRF (PANalytical Minipal 4), SEM-EDX (Hitachi SU3500), and FTIR (Thermo Scientific).

2.2. Procedures

2.2.1. Preparation

First, the preparation of raw materials, dolomite lime ($CaMg(CO_3)_2$), was analyzed using XRF to determine its composition. The analyzed dolomite lime was weighed to 10 grams. Dolomite dissolution was carried out using 30 ml of 85% phosphoric acid, then diluted with distilled water to reach a concentration of 5 N. The dissolution process was conducted with a magnetic stirrer at 30°C and a stirring speed of 450 rpm for 30 minutes reaction time. This process produced a mixture containing $CaHPO_4$, $MgHPO_4$, H_2O, and CO_2 gas.

2.2.2. Composite Formation

A 24 N NH_4OH solution was added to the dissolution mixture containing $CaHPO_4$, $MgHPO_4$, and H_2O until the pH reached 8. The precipitation process was conducted using a magnetic stirrer at 30°C and a stirring speed of 450 rpm for 15 minutes reaction time. The precipitation resulted in white crystalline solids, which were then subjected to filtration. The resulting precipitate, identified as the Ca-Mg-NH_4-PO_4 composite, was dried to eliminate water content. Drying was performed in an oven at 100°C for approximately 30-60 minutes.

Figure 1. The appearance of the Ca-Mg-NH_4-PO_4 composite from dolomite lime synthesis

2.2.3. Characterization

The characterization of the material involved several analytical techniques. The crystal structure of the samples was analyzed using XRD. Measurements were made with a Cu-Kα target anode (λ = 1.54060 Å) over a 2θ range of 10.0084° - 89.9764°, a step size of 0.0170°, and operating conditions of 40 kV and 30 mA. The elemental composition was analyzed using the XRF instrument. Morphological characteristics were analyzed using SEM, and elemental composition was analyzed through EDX. SEM imaging was conducted at magnifications of 1000×, 2500×, 3000× and 5000×, while EDX was performed at an operating voltage of 15 kV. The functional groups present in the samples were analyzed using FTIR, covering a wavenumber range of 1000 to 4000 cm^{-1}.

3. Results and Discussion

3.1. Synthesis of the Ca-Mg-NH_4-PO_4 Composite

The synthesis of the Ca-Mg-NH_4-PO_4 composite was achieved through the dissolution of dolomite lime in a 5 N phosphoric acid solution. During the dissolution process, observations revealed the formation of gas bubbles when the dolomite material reacted with the phosphoric acid solvent. The presence of gas bubbles indicated that the dissolution reaction of dolomite released CO_2 gas from the decomposition of $CaCO_3$. This observation is consistent with previous studies that report the production of CO_2 gas during the reaction of $CaCO_3$ with acid [29]. Following dissolution, precipitation was performed using an NH_4OH solution, forming a solid-phase product characterized by a powdered morphology and white color, as illustrated in Figure 1. The total mass of the synthesized product amounted to 20.7620 grams. To assess the success of the synthesis, the Ca-Mg-NH_4-PO_4 composite was analyzed using XRD, XRF, SEM-EDX, and FTIR techniques.

3.2. Characterization

3.2.1. XRD Analysis

The crystallographic characteristics of the Ca-Mg-NH_4-PO_4 composite were analyzed using XRD to determine its crystallinity and phase composition. The diffraction patterns provided valuable insights into the arrangement and purity of the composite, enhancing the overall understanding of its structural characteristics.

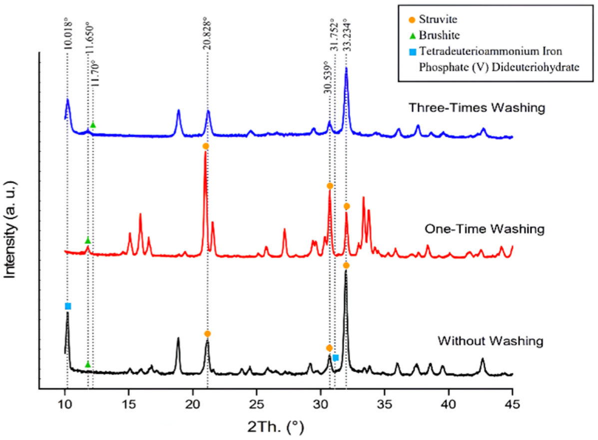

Figure 2. XRD diffraction patterns of Ca-Mg-NH_4-PO_4 composite

Figure 2 presents the composite's crystal composition under three washing treatments: untreated, single washing, and triple washing, as determined by XRD analysis. The diffraction patterns obtained from the study reveal distinct crystal formation and purity variations across each treatment condition. In the untreated composite, struvite was the dominant phase, constituting 42% of the composition at 2θ positions of 20.828°, 30.539°, and 33.234°. Brushite crystals represented 29% of the composition at a 2θ position of 11.650°. Additionally, tetradeuterioammonium iron phosphate (V) dideuteriohydrate crystals were detected as impurities, comprising 29% of the composition at 2θ positions of 10.018° and 31.752°. These impurity crystals are known to occur during the synthesis process.

Following a single washing treatment, impurity levels decreased significantly, increasing struvite content to 81% at 2θ positions of 20.828°, 30.539°, and 33.234°, while brushite accounted for 19% at 11.650°. The absence of impurities highlights the efficacy of washing in enhancing compositional purity. In the three-times washing treatment, brushite crystals were exclusively observed, comprising 100% of the composition at 11.70°. The diffraction pattern shows a more sloping peak with three washing times, with struvite no longer detected. The repetitive washing process led to the selective dissolution of ions, thereby removing struvite. Hence, it can be concluded that washing significantly influences the composition of the Ca-Mg-NH_4-PO_4 composite.

The XRD analysis results confirmed the orthorhombic structure of struvite and the monoclinic structure of brushite in the composite, aligning with prior research findings. Specifically, the highest crystal peak for struvite was observed at 2θ positions of 20.76°, 30.54°, and 32.86° (JCPDS no. 71-2089), while brushite exhibited its highest crystal peak at a 2θ position of 11.70° (JCPDS no. 72-0713) [34]. The XRD analysis provides valuable insights into the composite's crystallinity structure and demonstrates the effectiveness of washing in removing impurities from the composite product. Further analysis of morphology and elemental composition is recommended to better understand the composite's structure, thereby reinforcing the findings

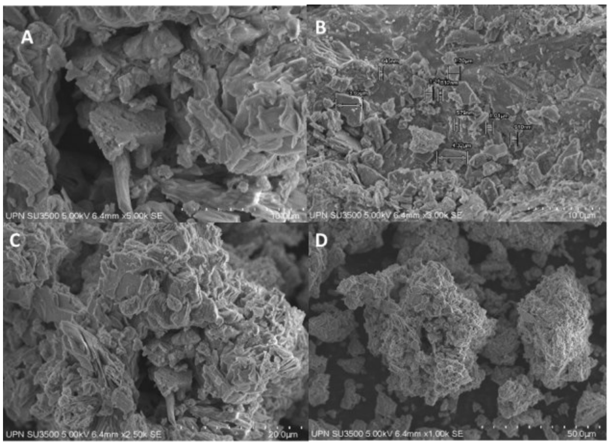

Figure 3. SEM images of Ca-Mg-NH_4-PO_4 composite at magnifications of (a) 5000×, (b) 3000×, (c) 2500×, and (d) 1000×

Table 1. XRF analysis results of Ca-Mg-NH_4-PO_4 composite

Component	Concentration (% wt)
CaO	22.2
MgO	11
P_2O_5	66.8

3.2.2. XRF Analysis

XRF analysis was conducted to determine the elemental composition of the Ca-Mg-NH_4-PO_4 composite product. The XRF analysis results in Table 1 indicated that the composite comprises several compounds such as CaO, MgO, and P_2O_5. The Ca and Mg elements within the composite are sourced from the dissolution of dolomite lime. At the same time, the phosphorus pentoxide (P_2O_5) is derived from the phosphoric acid solution utilized as a solvent for dolomite lime. The most significant content of the Ca-Mg-NH_4-PO_4 composite constituent is the P_2O_5 compound at 66.8%, followed by CaO at 22.2% and MgO at 11%. The predominance of P_2O_5 highlights the composite's potential application as a phosphate-based fertilizer. The absence of detectable NH_4 compounds in the XRF analysis is due to the difficulty of this method in identifying non-metallic ions, such as nitrogen. To overcome this limitation, further elemental analysis was conducted using EDX, which enabled the quantification of nitrogen within the Ca-Mg-NH_4-PO_4 composite product.

3.2.3. SEM Analysis

The morphology of the Ca-Mg-NH_4-PO_4 composite was analyzed using SEM to gain insights into its structural characteristics. As illustrated in Figure 3, the composite predominantly exhibits a layered, slab-like structure indicative of brushite crystal formation [35]. This is due to the presence of Ca in the composite product. The presence of Ca ions in the composite significantly influences the growth dynamics of the crystal compounds. Specifically, Ca ions facilitate the closure of gaps between crystals, which may restrict the collision of crystal nuclei and inhibit the formation of struvite, resulting in an imperfect crystalline structure [36]. The composite displayed a predominantly amorphous morphology, likely due to impurities introduced during the synthesis process. At a magnification of 3000×, SEM imaging revealed crystal sizes within the composite ranging from 4.32 µm to 579 nm. SEM analysis provides detailed information about the surface morphology of the composite.

3.2.4. EDX Analysis

As shown in Figure 4, EDX analysis confirmed the presence of the critical elemental constituents, such as Ca, Mg, N, P, and O, in the Ca-Mg-NH_4-PO_4 composite synthesized from dolomite. The elemental composition (%) of the Ca-Mg-NH_4-PO_4 composite meets the quality standards for phosphate-based multi-nutrient fertilizers, ensuring its suitability for agricultural applications [13]. While minor impurities, such as Al at 0.26%, were detected, this concentration remains well below the harmful threshold of 30%, indicating that the composite can be safely applied to plants without adverse effects [37]. The ability of EDX to identify nitrogen, a crucial nutrient for plant growth, addresses the limitations of XRF analysis, which is often inadequate for detecting non-metallic elements. Collectively, EDX and XRF analysis provide a comprehensive understanding of the composite's overall composition, reinforcing its potential as a multi-nutrient fertilizer.

3.2.5. FTIR Analysis

FTIR analysis was conducted to identify functional groups in the Ca-Mg-NH_4-PO_4 composite, with the resulting spectrum displayed in Figure 5. Significant peaks were observed at 949 cm^{-1}, 972 cm^{-1}, 1027 cm^{-1}, 1432 cm^{-1}, and 1466 cm^{-1}, corresponding to important chemical vibrations. The wavenumber at 949 cm^{-1} and 972 cm^{-1} indicate the P-O-C stretching vibrations of aromatic phosphate, which are thought to be formed during the dissolution of raw materials with phosphoric acid. The peak at 1027 cm^{-1} corresponds to the P-O-C stretching vibration of aliphatic phosphate, indicating the presence of phosphate ions in the composite.

The peak at 1432 cm^{-1} is associated with asymmetric C-H bending vibrations of aliphatic groups [38], while the peak at 1466 cm^{-1} corresponds to the N-H bending vibration of amine groups, likely formed through the addition of ammonium hydroxide as a precipitating agent [39]. These functional groups, particularly the P-O-C and N-H bonds, substantiate the composite's phosphate and nitrogen content, validating its potential role as a multi-nutrient fertilizer. Furthermore, FTIR analysis underscores the importance of these functional groups in ensuring nutrient stability within the composite matrix, enhancing its efficacy in agricultural applications.

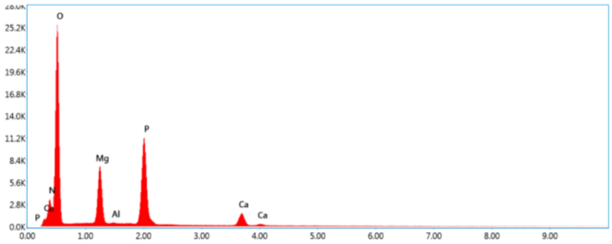

Figure 4. Results of EDX analysis of Ca-Mg-NH_4-PO_4 composite

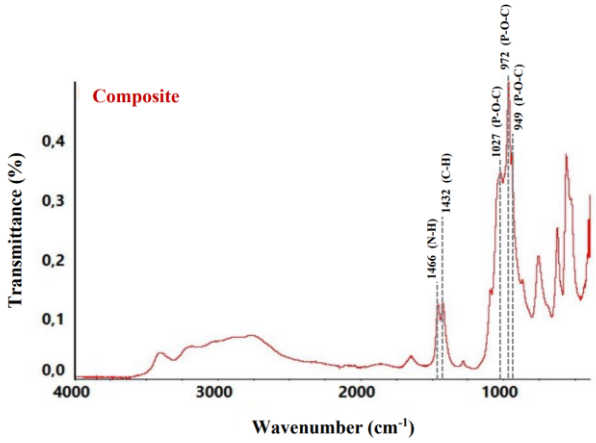

Figure 5. Results of FTIR analysis of Ca-Mg-NH_4-PO_4 composite

4. Conclusion

Based on the research results, it can be concluded that the synthesis of Ca-Mg-NH_4-PO_4 composite by the precipitation method has been successfully carried out, as evidenced by the results of XRD, SEM-EDX, XRF, and FTIR analysis. XRD characterization showed the formation of struvite crystals at 2θ = 20.828°; 30.539°; 33.234°, brushite crystals at 2θ = 11.650°, and impurity compounds that appeared during the synthesis process. SEM analysis indicated that the composite consists of monoclinic crystals with a crystal size ranging from 4.32 µm to 579 nm. XRF and EDX analysis confirmed that the composite contains Ca-Mg-NH_4-PO_4 components. FTIR analysis revealed the presence of P-O-C groups in the wavenumber range of 949 cm^{-1}, 972 cm^{-1}, and 1027 cm^{-1}, and N-H groups at 1466 cm^{-1}. Based on these analytical results, the Ca-Mg-NH_4-PO_4 composite meets the quality standards as a phosphate-based multi-nutrient fertilizer for plants.

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