ISSN 2355-8776

Synthesis and Characterization of Calcium Magnesium Phosphate ($CaMg(HPO_4)_2$) Nanoparticle Material Made from Dolomite

Ade Naufal [a,1,*], Rangga Putra Adji Wibisana [a,2], Ketut Sumada [a,3], Caecilia Pujiastuti [a,4], Susilowati [a,5], Srie Muljani [a,6]

[a] Department of Chemical Engineering, Faculty of Engineering and Science, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Surabaya, Indonesia
[1] naufal.ade50@gmail.com*; [2] dakrondektor1927@gmail.com; [3] sumadaketut@gmail.com; [4] caecilia.tk@upnjatim.ac.id; [5] zuzisukasno@gmail.com; [6] sriemuljani.tk@upnjatim.ac.id
* corresponding author

ARTICLE INFO

Article history
Received July 02, 2025
Revised July 19, 2025
Accepted July 21, 2025

Keywords
Dolomite
Extraction
Multinutrient fertilizer
Nanomaterial
Precipitation

ABSTRACT

Dolomite is a mineral with potential as an alternative material to enhance fertilizer quality. Traditional fertilizers often suffer from low nutrient use efficiency, as much of the applied nutrients are lost before being absorbed by plants, leading to economic and environmental losses. Nanofertilizers offer a solution by increasing nutrient uptake efficiency due to their small particle size and large surface area. This study aims to characterize calcium magnesium phosphate nanoparticles synthesized from dolomite and evaluate their elemental solubility in water. The synthesis involved dissolving dolomite at five different weights (20, 30, 40, 50, 60 grams) and reacting it with varying volumes of disodium phosphate (400–1200 mL). SEM analysis revealed a thin plate or needle-like crystal structure. The best composition—20 g dolomite and 1200 mL disodium phosphate—resulted in a product containing 22.13% calcium and 19.57% phosphorus, with an average Ca/P ratio of 1.005, as shown by EDX. Magnesium was not present in the precipitate due to the synthesis pH (4–6), which is below the optimum pH (8–9.5) for magnesium precipitation. BET analysis confirmed a particle size of 96 nm, classifying the material as a nanomaterial. Solubility tests suggest the material functions as a slow-release fertilizer. Given its calcium and phosphorus content and nanoscale structure, this material shows strong potential as a high-quality, multinutrient nanofertilizer that can improve nutrient absorption and support sustainable agriculture.

1. Introduction

Dolomite is a sedimentary rock naturally occurring in an environment and composed mainly of calcium carbonate ($CaCO_3$), magnesium carbonate ($MgCO_3$), and other oxide compositions. The chemical formula of dolomite is $CaMg(CO_3)_2$ [1]. Indonesia, a country located on the equator, has a long history of carbonate minerals. The data from the Minister of Energy and Mineral Resources of the Republic of Indonesia shows that Indonesia has measured resources of dolomite rock of more than 280 million tons by the end of 2023 [2]. Generally, dolomite is applied directly to the agricultural, plantation, and pond sectors to overcome soil with high acidity levels because dolomite has the advantage of maintaining high soil pH. In addition to direct application, dolomite can first be processed with process technology to increase its economic value. The potential for processing dolomite minerals includes the dissolution process with acid, the separation process of calcium and magnesium, the calcination process, and the material synthesis process with chemical reactions [3].

Extraction is a process in chemical engineering that involves the separation of specific components from raw materials [4]. In this study, extraction is used to dissolve calcium and magnesium from dolomite using an acetic acid solution [5]. After the extraction process, the calcium and magnesium dissolved in calcium magnesium acetate will react with disodium phosphate in the precipitation process [6]. Precipitation is the process of forming a solid material from a solution through a chemical reaction. A solid substance is created from a solution by one of two mechanisms: the material is converted into an insoluble, or the solvent composition is modified to reduce the solubility of the substance in it [7].

Nanomaterials are materials with particle sizes on the nanometer scale, namely between 1 and 100 nanometers, and contain various properties like small diameters, low weight, and high surface area [8]. Nanotechnology is a scientific discipline that allows us to understand and manipulate matter at the atomic and molecular level, and its application in agriculture has the potential to address critical challenges faced by Indonesia [9]. The use of nanoparticles in agriculture has recently been introduced, such as nanofertilizer [10], which has shown its effectiveness in plants to help increase nutrient absorption due to their small particle size, resulting in a larger surface area and reducing the need for excessive fertilizer [11]. The nutrient use efficiency of traditional fertilizers is very low. It has been reported that approximately 40–70% of nitrogen, 80–90% of phosphorus, and 50–90% of potassium from applied fertilizers are lost in the environment and do not reach plants, resulting in significant economic losses. To address this scenario, agriculture must adopt more advanced technologies, practices, and methods, including the use of nanofertilizers [12].

In this study, the first synthesis of calcium magnesium phosphate nanoparticle material from Indonesian dolomite will be carried out. This synthesis technology is expected to increase the economic value of dolomite by utilizing the calcium and magnesium content of dolomite to react with phosphate to obtain calcium magnesium phosphate material needed as a multinutrient fertilizer. The nanoparticle material can also increase contact between fertilizer and soil and plant roots, thereby accelerating the dissolution and release of nutrient ions. We hypothesize that acetate-mediated synthesis yields stable CaMg(HPO4)2 nanoparticles. Acetate solution can be used as an option for the synthesis of nanoparticle material products. Acetate ions can be stabilizers that interact with the surface of nanoparticles, creating a protective layer that prevents nanoparticles from approaching each other and agglomerating into microparticles [13]. Prior studies [14] synthesized calcium magnesium ammonium phosphate fertilizer and failed to synthesize a nanofertilizer, achieving only a size of 579 nm.

This study aims to determine the characteristics of calcium magnesium phosphate nanoparticle material and investigate the effect of the solubility of elements in this material on water. Dissolution in water can provide an early indication of the availability of nutrients after fertilizer is applied to the soil and exposed to water, such as rainwater or irrigation. Elements that dissolve quickly in water are also likely to be more rapidly available to plants in the soil.

2. Research Methodology

2.1. Materials

The primary material used in this study is Indonesian dolomite, which was purchased from PT. Agro Nusantara Indonesia. Other materials needed are acetic acid / CH_3COOH (99% purity), disodium phosphate / Na_2HPO_4 (Tech. grade), and distilled water. All three were purchased from CV. Jaya Makmur Kimia.

2.2. Procedures

1) Dolomite Pre-Treatment

Dolomite raw materials were analyzed using X-Ray Fluorescence (XRF) test to determine their composition and X-Ray Diffraction (XRD) test to determine their crystal structure.

2) Synthesis Process

The synthesis of $CaMg(HPO_4)_2$ nanoparticle material includes two processes: the dissolution of dolomite, which contains calcium and magnesium components, using acetic acid solvent, and the precipitation of calcium magnesium acetate solution by adding disodium phosphate solution to form calcium magnesium phosphate precipitate. The reactions that occur are as follows:

$$CaMg(CO_3)_{2\,(s)} + 4CH_3COOH_{(aq)} \rightarrow [CaMg(CH_3COO)_4]_{(aq)} + 2CO_{2\,(g)} + 2H_2O_{(aq)} \quad [1]$$

$$[CaMg(CH_3COO)_4]_{(aq)} + Na_2HPO_{4\,(aq)} \rightarrow CaMg(HPO_4)_{2\,(s)} + 4CH_3COONa_{(aq)} \quad [2]$$

Dolomite was weighed using an analytical balance according to the variables run (20, 30, 40, 50, 60) grams. The weighed dolomite was then dissolved with 250 ml of 40% acetic acid at a stirring speed of 200 rpm for 60 minutes. Stirring speed of 200 rpm was chosen because it is included in the effective conditions for dissolving calcium and magnesium [15] and the stirring speed conditions are available on the lab stirrer used in the dissolution process.was chosen because it is included in the effective conditions for dissolving calcium and magnesium and the stirring speed conditions are available on the lab stirrer used in the dissolution process. The dissolution results were then filtered with filter paper with a maximum pore size of 20 μm to obtain the filtrate. The filtrate was added with 1,5 N disodium phosphate solution according to the variables run (400, 600, 800, 1000, 1200) ml, then stirred until homogeneous and precipitated for 30 minutes. Disodium phosphate concentration is based on the stoichiometry of the reaction of the need for disodium phosphate to react with metal ions. The precipitation results were filtered with filter paper to obtain the $CaMg(HPO_4)_2$ material precipitate, then dried in a laboratory oven for 3 hours at a temperature of 110 °C to remove water and other easily volatilized impurities [16]. The dried $CaMg(HPO_4)_2$ material was then weighed using an analytical balance.

3) Dissolution Test

The process of dissolving calcium magnesium phosphate nanoparticle material was carried out to determine the solubility of ions in the $CaMg(HPO_4)_2$ material in water. The results before and after dissolution will be compared with Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDX) analysis using a Hitachi SU3500 SEM. The products formed will be divided into two groups for SEM-EDX analysis before and after dissolution. The material dissolution process was carried out by dissolving the $CaMg(HPO_4)_2$ material with water and stirring for 10 minutes. The solution results were filtered using filter paper and obtained $CaMg(HPO_4)_2$ precipitate after dissolution, which was then dried in an oven for 3 hours at a temperature of 110 °C. The dried $CaMg(HPO_4)_2$ material was subjected to SEM-EDX analysis.

4) Characterization Techniques

Characterization of the calcium magnesium phosphate nanoparticle material formed includes several analyses. The formed product was analyzed using the Brunauer-Emmett-Teller (BET) test with a 2000-16 Quantachrome Instrument to determine the specific surface area that can be correlated with the size of the nanoparticle material [17]. Morphological characterization of the product was analyzed using Scanning Electron Microscopy (SEM) [18]. The chemical element composition was analyzed using Energy Dispersive X-ray Spectroscopy (EDX) [19].

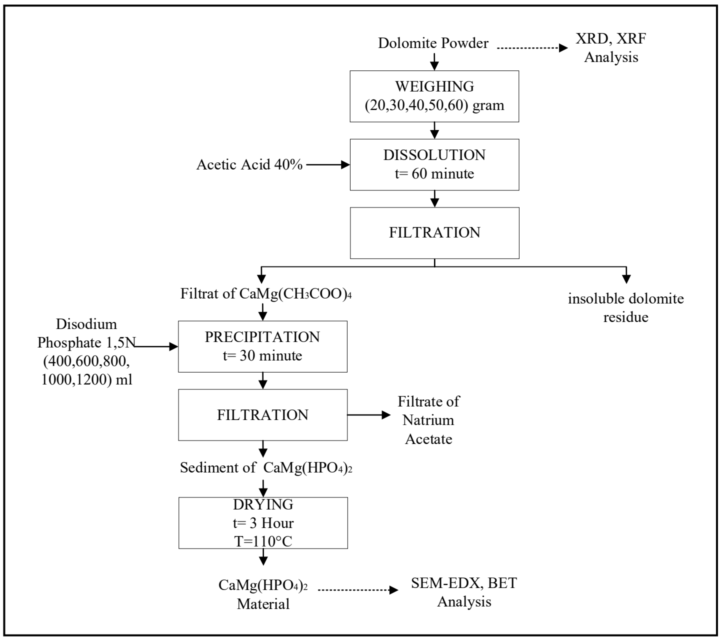

Fig. 1.$CaMg(HPO_4)_2$ Nanoparticle Material Synthesis Flow Chart

3. Results and Discussion

3.1. Raw Material Characterization and Result of Synthesis

The dolomite raw material is first subjected to X-Ray Diffraction (XRD) analysis to determine its crystal structure and X-Ray Fluorescence (XRF) analysis to determine the content contained in the dolomite.

Table 1. Crystal Structure of Dolomite

Crystal Name	Chemical Formula	Percent (%)
Dolomite	$CaMg(CO_3)_2$	98.82
Quartz	SiO_2	0.39
Magnesite	$MgCO_3$	0.19
Pyrite	FeS_2	0.11
Anorthite	Ca_3SiO_5	0.1
Calcite	$CaCO_3$	0.01
Siderite	$FeCO_3$	0.01

X-Ray Diffraction (XRD) analysis to determine its crystal structure. Based on Table 1, the dominant crystal structure found in the raw material is dolomite crystal with a percentage of 98.82%. Other crystals found in the raw material are quartz, magnesite, pyrite, anorthite, calcite, and siderite crystals, each with a percentage of less than 1%.

Table 2. Compound Content of Dolomite

Chemical Formula	Percent (%)
CaO	34.75
MgO	20.42
SiO_2	0.36
Fe_2O_3	0.21
Al_2O_3	0.19
Na_2O	0.06
K_2O	0.02

X-Ray Fluorescence (XRF) analysis to determine the chemical composition or content contained in dolomite. It was found that the calcium oxide (CaO) content was 34.75% and magnesium oxide (MgO) was 20.42% which was used as raw material. The presence of impurities in tiny amounts, below 1%, does not have a significant effect on the calcium and phosphate levels in the final product. According to [20], the impurities have no significant impact on the content of nitrogen and phosphorus in urea phosphate products. Otherwise, if the impurity content is higher, it may affect both the process and the final product. According to [21], the influence of impurities Al_2O_3 and Fe2O3 of more than 3% can affect the filtration rate and post precipitation issues.

Table 3. Result of Synthesis Nanoparticle Material $CaMg(HPO_4)_2$

Dolomite Weight (gram)	Volume of Disodium Phosphate 1,5 N (ml)	Calcium Content (%)	Phosphorus Content (%)	Ca/P Ratio	Particle Size (nm)
20	400	28.46	18.27	1.2073	169.42
	600	26.88	18.59	1.1202	151.08
	800	25.29	18.92	1.0361	132.73
	1000	23.71	19.24	0.9549	114.39
	1200	22.13	19.57	0.8764	96.05
30	400	27.60	18.14	1.1792	169.30
	600	26.25	18.55	1.0967	151.88
	800	24.90	18.96	1.0178	134.47
	1000	23.55	19.37	0.9422	117.06
	1200	22.20	19.78	0.8698	99.65
	400	26.74	18.01	1.1507	169.23
	600	25.63	18.51	1.0731	152.29

Dolomite Weight (gram)	Volume of Disodium Phosphate 1,5 N (ml)	Calcium Content (%)	Phosphorus Content (%)	Ca/P Ratio	Particle Size (nm)
40	800	24.51	19.00	0.9996	135.34
	1000	23.39	19.50	0.9298	118.39
	1200	22.27	19.99	0.8634	101.45
	400	26.21	17.55	1.1574	169.14
	600	25.24	18.21	1.0742	152.89
50	800	24.28	18.88	0.9967	136.65
	1000	23.31	19.54	0.9246	120.40
	1200	22.35	20.20	0.8572	104.15
	400	25.68	17.09	1.1645	169.05
	600	24.38	18.42	1.0255	147.82
60	800	23.72	19.09	0.9633	126.58
	1000	23.07	19.75	0.9053	116.72
	1200	22.42	20.42	0.8510	106.85

3.2. Analysis of Chemical Composition and Morphology of $CaMg(HPO_4)_2$ Material by Scanning Electron Microscopy Energy Dispersive - X-ray Spectroscopy (SEM-EDX)

The morphological characterization of the product was analyzed using Scanning Electron Microscopy (SEM), while the chemical element composition was analyzed using Energy Dispersive X-ray Spectroscopy (EDX).

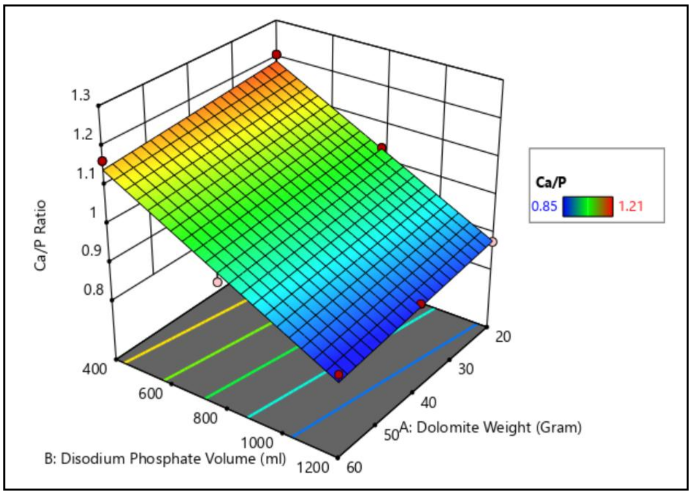

Fig. 2. Effect of Dolomite Weight and Disodium Phosphate Volume on Ca/P Ratio

Fig.2 is a 3D surface plot showing the effect of dolomite weight and disodium phosphate volume on the Ca/P ratio. The weight of dolomite used varies from 20 g to 60 g, with a range of 40 g, while the volume of disodium phosphate used varies from 400 ml to 1200 ml, with a range of 800 ml. The colors represent the Ca/P ratio, where red indicates the highest Ca/P ratio and blue indicates the lowest Ca/P ratio. Increasing the weight of dolomite will increase the calcium content and increase the Ca/P ratio, while increasing the volume of disodium phosphate will increase the phosphate content and decrease the Ca/P ratio. According to [22], in conditions of excess phosphate ions, the system tends to form calcium phosphate compounds that are rich in phosphate with a lower Ca:P ratio, such as brushite or dicalcium phosphate dihydrate, which results in an increase in phosphorus content in the sediment and a relative decrease in calcium content.

Based on EDX analysis, the chemical composition of the $CaMg(HPO_4)_2$ material is oxygen (O), phosphorus (P), calcium (Ca), sodium (Na), and aluminum (Al) ions. Magnesium ions that should be available in the product were not identified. The dominant oxygen ions are mostly bound to calcium and phosphorus ions as the main products, and a small part is bound to sodium ions that form sodium acetate byproducts that are included in the main product because the product washing process was not carried out. The small amount of aluminum ions detected is possible due to dolomite impurities that are also precipitated, which does not affect the product.

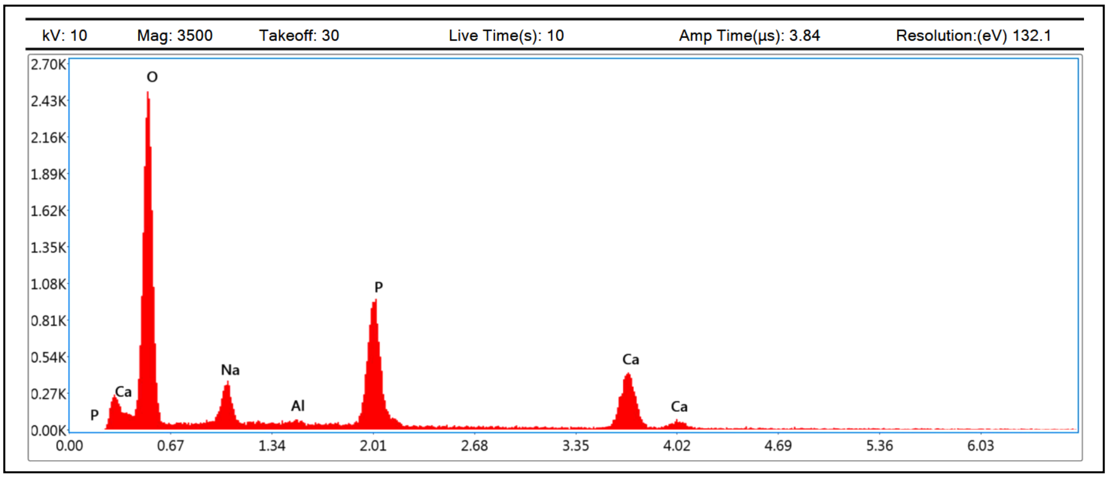

Fig. 3.EDX Analysis Results of CaMg(HPO4)2 Material

Mg ions are not precipitated with the product and remain dissolved with the byproducts. This is because the synthesis process conditions with disodium phosphate volume from 400 ml to 1200 ml only reach a pH range between 4 and 6. According to [23], the optimum pH range for magnesium precipitation is pH 8-9.5. Increasing pH in the precipitation process is crucial for affecting the results of the products obtained. Increasing pH can be done by adding sodium hydroxide.

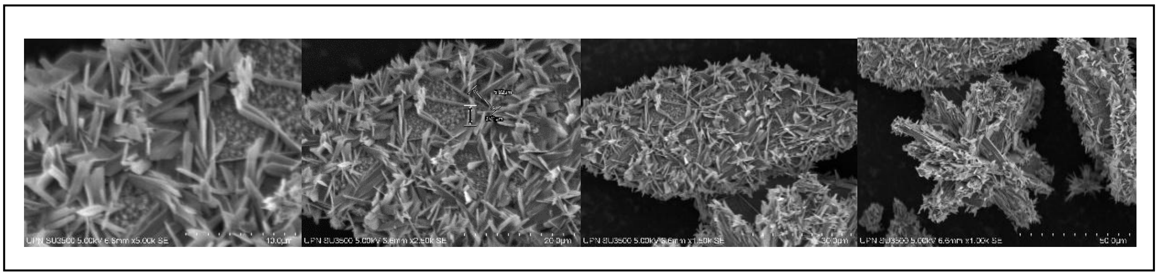

Fig. 4.SEM of $CaMg(HPO_4)_2$ Material in Dolomite Weight Condition of 30 grams and Disodium Phosphate Volume of 600 ml with Magnification of 5000x, 2500x, 1500x, and 1000x

The morphology of $CaMg(HPO_4)_2$ material was analyzed using SEM analysis to determine its structural characteristics. As seen in Figure 4, the material shows a crystalline structure in the form of thin plates or needles. At a magnification of 5000x, SEM imaging revealed crystal size within the range from 3.2 µm to 10 µm. In this research [24], synthesized hydroxyapatite crystals with different morphologies, including plate-like, hexagonal prism, and needle-like. These crystals grow randomly and stack on each other, exhibiting irregular crystal growth. The presence of impurities can affect crystal growth, for example, resulting in a longer crystal shape or morphological distortion. SEM analysis provides detailed information about the surface morphology of $CaMg(HPO_4)_2$ material. Based on EDX, the absence of Mg means the product only contains calcium and phosphate. From the average of calcium and phosphorus content, it was found that the Ca/P ratio of the material produced was 1.005, so that the material was identified as calcium phosphate with the type monetite or brushite [25]. The morphological form of the product based on SEM analysis, according to research [26], is that brushite is in the form of thin and rectangular plates.

3.3. Ion Solubility in $CaMg(HPO_4)_2$ Material Using Energy Dispersive X-ray Spectroscopy (EDX) Analysis

The $CaMg(HPO_4)_2$ product was dissolved in water to see the solubility of ions in the $CaMg(HPO_4)_2$ material. According to the weight percent, the calcium and phosphorus elements did not decrease after dissolution. This indicates that the $CaMg(HPO_4)_2$ product is not readily soluble in water. Data from [27] shows that the solubility of dicalcium phosphate in water is 0.02 g/100 g of water, so it is classified as difficult to dissolve. Oxygen and sodium ions decreased after dissolution because the sodium ions here come from the sodium acetate byproduct, which is included in the main product, due to the lack of a product washing process. By the sodium acetate solubility data from [27], the solubility of sodium acetate in water is 46.5 g/100 g of water, so it is classified as easily soluble. According to [28], washing the formed product precipitate was carried out to remove the filtrate from the resulting by-product. Therefore, after washing, the sodium ion decreased. The oxygen ions that experience a decrease are those bound to sodium acetate. For aluminum ions that increase after dissolution, this increase may be due to impurities in the water used in the dissolution process of the $CaMg(HPO_4)_2$ product. Calcium and phosphorus ions in the $CaMg(HPO_4)_2$ product are difficult to dissolve in water, so that the composition of calcium and phosphorus before and after dissolution is not much different. Still, based on the weight percent of calcium and phosphorus ions, they increase after dissolution. This is because the weight percent is the ratio of all elements, where the natrium and oxygen elements decrease, so the weight percent of calcium and phosphorus ions automatically increases. After dissolution, there are still many oxygen ions. A small part of these ions is bound to sodium because the sodium acetate byproduct has not completely dissolved. Most of the ions are bound to the $CaMg(HPO_4)_2$ product, making it difficult to dissolve in water. The nature of the $CaMg(HPO_4)_2$ material, which is difficult to dissolve in water, allows the material to become a slow-release fertilizer. The advantages of slow-release fertilizer, as mentioned [11], make the product have advantages compared to other commercial fertilizers, such as DAP fertilizer, where the solubility of DAP fertilizer is 58.8 g/L, which is considered a highly soluble fertilizer [29]. The solubility of calcium and phosphate ions is influenced by pH, where solubility decreases as the pH approaches neutral conditions. Furthermore, at a higher pH of around 8.5, dissolved calcium and phosphate ions will trigger the formation of new phases such as hydroxyapatite [30]. It is known that the pH of the dissolution medium is 6.2, which supports partial dissolution of brushite without inducing phase transformation. Brushite can be a slow-release phosphate fertilizer, particularly suitable for acidic to neutral soils [31]. Under such conditions, it can gradually release nutrients over several weeks, reducing phosphate runoff and improving nutrient use efficiency compared to highly soluble fertilizers like DAP.

Table 4. Chemical Element Composition of $CaMg(HPO_4)_2$ Material

Before Dissolution			**After Dissolution**		
Element	***% Weight***	***% Atomic***	***Element***	***% Weight***	***% Atomic***
O	54.75	71.85	O	43.91	62.75
Na	3.83	3.5	Na	2.74	2.73
Al	1.25	0.98	Al	2.42	2.05
P	17.09	11.59	P	18.1	13.36
Ca	23.07	12.09	Ca	31.76	18.11

3.4. Analysis of $CaMg(HPO_4)_2$ Material Particle Size Using the Brunauer-Emmett-Teller (BET) Method

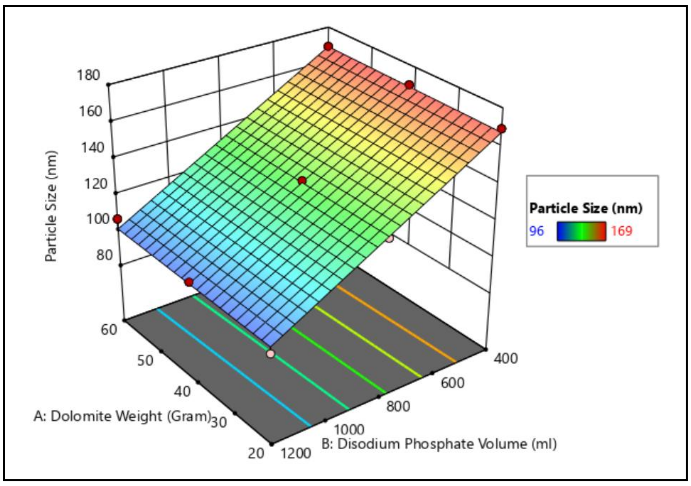

Fig. 5. Effect of Disodium Phosphate Volume on Particle Size

Fig.5 is a 3D surface plot showing the effect of dolomite weight and disodium phosphate volume on the particle size obtained. The weight of dolomite used varies from 20 g to 60 g, with a range of 40 g, while the volume of disodium phosphate used varies from 400 ml to 1200 ml, with a range of 800 ml. The colors represent the particle size, with red indicating the largest and blue the smallest.

Increasing the weight of dolomite will increase the concentration of Ca^{2+} ions in the solution, which encourages crystal growth more dominantly than nucleation. According to [32], the rate of ion association that forms larger particles increases by increasing the concentration of metal ions and according to [33], the more phosphate volume added, the more it will affect the pH to be more alkaline, thereby increasing the nucleation rate which results in much more particle nuclei and will trigger the formation of smaller particles.

4. Conclusion

Dolomite mineral can be used as a raw material for making $CaMg(HPO_4)_2$ nanoparticle material with a dissolution process using acetic acid and a precipitation process using disodium phosphate solution. At a dolomite weight of 20 grams and a disodium phosphate volume of 1200 ml, the chemical composition obtained was Ca of 22.13% and P of 19.57% with an average Ca/P ratio of 1.005. The particle size of the $CaMg(HPO_4)_2$ material was 96 nm, so that the product was classified as a nanomaterial. The material shows potential as a slow-release fertilizer based on the solubility test. Further research should focus on optimizing pH for mg formation, long-term soil fertility, and large-scale applications.

Acknowledgment

The authors express their gratitude to the Department of Chemical Engineering, Faculty of Engineering and Science, University of Pembangunan Nasional "Veteran" Jawa Timur, Surabaya, Indonesia, for its support. This assistance has been invaluable in advancing this research and fostering the authors' creativity.

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