International Journal of Eco-Innovation in Science and Engineering Vol.07 (1), 2026 (39-46)
https://ijeise.upnjatim.ac.id/ E-ISSN: 2721-8775

Article

Synthesis of Ca–Si–K–P Composite from Carbide Lime and Rice Husk using Precipitation Method

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Received: 01st April 2026; Revised: 07th April 2026; Accepted: 25th May 2026;
Available online: 30th May 2026; Published regularly: May and November

Abstract

The development of compositionally multinutrient composite offers a promising pathway to overcome the limitations of conventional fertilizer systems that predominantly focus on N–P–K. In this study, a Ca–Si–K–P composite was synthesized via a controlled precipitation route utilizing carbide lime waste and rice husk ash as sustainable precursors. The effects of precipitation pH (7–11) and calcination temperature (600–1000 °C) on oxide composition and yield were systematically investigated. The results demonstrate that increasing pH from 7 to 11 significantly enhances CaO content, reaching its optimum at pH 9–11, while SiO_2 content decreases by up to ~20–30% under highly alkaline conditions due to increased silicate solubility. The K_2O fraction remains relatively low (<10 wt%) across all conditions, primarily due to dissolution losses and thermal volatilization, whereas P_2O_5 exhibits minor variation (<5 wt%) within the studied pH range. Increasing calcination temperature from 600 to 1000 °C leads to a relative increase in SiO_2 content by approximately 10–15%, accompanied by a decrease in CaO fraction and partial loss of K_2O and P_2O_5 at temperatures ≥900 °C. The product yield exceeds 100% due to KOH addition during pH adjustment and shows a decreasing trend with temperature, dropping by approximately 10–20% from 600 to 1000 °C as a result of dehydration and decarbonation processes. Overall, alkaline precipitation conditions (pH 9–11) combined with moderate calcination temperatures (700–800 °C) provide the most favorable balance between compositional homogeneity and yield. These findings highlight the potential of waste-derived resources and precipitation engineering in producing composition controlled Ca–Si–K–P composites, offering significant prospects for application as advanced multinutrient fertilizer precursors.

Keywords: Ca–Si–K–P composite, controlled precipitation, waste valorization, compositional engineering, multinutrient fertilizer

1. Introduction

The increasing global demand for food, coupled with land degradation and declining soil fertility, necessitates the development of more efficient and sustainable fertilization strategies [1]. While conventional fertilizer systems primarily emphasize nitrogen–phosphorus–potassium (N–P–K) formulations [2], other essential macronutrients such as calcium (Ca) and silicon (Si) are frequently supplied separately or incorporated without deliberate chemical integration[3]. From a compositional standpoint, Ca in stabilizing cell walls and maintaining membrane integrity, and Si enhances resistance to abiotic and biotic stresses, are often not integrated into a single chemical engineering system. Theoretically, Ca contributes to structural

DOI:10.4186/ijeise.upnjatim.ac.id/E-ISSN: 2721-8775

reinforcement and physiological regulation [4], P is central to energy transfer and root development, K regulates osmotic balance and enzyme activation [5], and Si strengthens plant tissues and improves water-use efficiency [6]. Integrating these four elements into a multicomponent Ca–Si–K–P system offers a compositional platform for the development of integrated multinutrient precursor systems.

From a materials chemistry perspective, the $CaO–SiO_2–K_2O–P_2O_5$ quaternary system exhibits complex phase interactions that govern structural stability, solubility, and nutrient release kinetics in soil environments. The formation of phases such as calcium phosphates, calcium silicates, potassium phosphates, and mixed Ca–Si–P compounds can significantly influence dissolution behavior and nutrient availability. Therefore, a synthesis approach capable of controlling chemical composition, compositional development, and microstructural homogeneity is crucial for designing advanced multinutrient fertilizer materials [7]. Chemical precipitation offers distinct advantages, including control over supersaturation, pH, and ionic interactions, enabling improved elemental distribution and more homogeneous phase formation compared with conventional solid-state mixing methods [8], [9]. In this framework, precipitation pH and calcination temperature are considered critical processing parameters, as variations in these variables are expected to directly affect the final oxide composition of the synthesized Ca–Si–K–P composite.

In line with sustainability and circular economy principles, the valorization of industrial and agricultural wastes as alternative raw materials has gained increasing attention. Carbide lime waste, which is rich in $Ca(OH)_2$, represents a potential low-cost calcium source [10], while rice husk, upon controlled combustion, yields silica-rich ash containing predominantly amorphous SiO_2. However, previous studies have largely focused on the individual utilization of these wastes or their partial incorporation into conventional fertilizer formulations. Furthermore, most multinutrient fertilizer developments rely on mechanical blending or simple granulation processes without chemical-level phase engineering, often resulting in heterogeneous nutrient distribution and uncontrolled release behavior.

The novelty of this study is synthesis of a multicomponent $CaO–SiO_2–K_2O–P_2O_5$ composite through a controlled precipitation approach using secondary resources, with emphasis on controlled elemental composition, compositional homogeneity, and chemical uniformity as a preliminary yet fundamental basis for the development of multinutrient fertilizer precursor materials.

2. Material and Method

2.1. Material

Carbide lime waste obtained from PT. Dwigasindo Abadi (Indonesia) was used as the calcium precursor. Based on previously reported data by Rattanashotinunt *et al.* (2013), carbide lime typically contains CaO (~56.6 wb%) and $CaCO_3$ (~26.8 wb%), along with minor oxides such as SiO_2 (~6.3 wb%), Al_2O_3 (~1.3 wb%), Fe_2O_3 (~0.6 wb%), and MgO (~0.1 wb%). These literature-reported values were adopted as a reference for stoichiometric calculations in the synthesis design. No independent compositional characterization of the raw carbide lime was conducted in the present study. Rice husk obtained from Mojokerto, East Java, Indonesia, was used as the silica precursor. After combustion, rice husk ash (RHA) was utilized for potassium silicate extraction. According to [11], RHA is predominantly composed of SiO_2 (~95.9 wt%) with minor inorganic impurities. The reported composition was used as a reference to estimate the silica contribution during synthesis. Phosphoric acid (H_3PO_4), potassium hydroxide (KOH), and distilled water were used as received without further purification.

2.2. Experimental Procedure

The Ca–Si–K–P composite was synthesized through a sequential precipitation steps consisting of tricalcium phosphate preparation, potassium silicate extraction, multicomponent precipitation, and calcination.

2.2.1. Preparation of Tricalcium Phosphate

DOI:10.4186/ijeise.upnjatim.ac.id/E-ISSN: 2721-8775

Tricalcium phosphate was prepared by dispersing 50 g of sieved carbide lime (100 mesh) in distilled water to obtain a 2 N $Ca(OH)_2$ suspension with a total volume of 674.8 mL. A 2.7 N H_3PO_4 solution (499.9 mL) was added gradually under continuous stirring to promote controlled precipitation. The reaction mixture was maintained for 24 h at room temperature to ensure sufficient interaction between calcium and phosphate species. The precipitated solid was separated and calcined at 700 °C for 1 h to obtain $Ca_3(PO_4)_2$ as the calcium–phosphate precursor.

2.2.2. Extraction of Potassium Silicate

Potassium silicate solution was obtained by mixing 9.0104 g of rice husk ash (100 mesh) with 20 wt% KOH solution in 67.3 mL of distilled water. The mixture was heated at 85 °C under continuous stirring at 100 rpm for 90 min to facilitate silica dissolution. After cooling to room temperature, the suspension was filtered to remove insoluble residues. The resulting filtrate, containing dissolved potassium silicate (K_2SiO_3), was used directly in the composite precipitation step without further purification.

2.2.3. Precipitation of Ca–Si–K–P Composite

The synthesized tricalcium phosphate (69.7722 g) was combined with the potassium silicate solution under continuous stirring to initiate composite formation. The pH of the system was adjusted using KOH solution to obtain five controlled conditions (pH 7, 8, 9, 10, and 11). The mixture was maintained under stirring to promote homogeneous distribution of calcium, silicate, potassium, and phosphate species. After precipitation, the solid product was filtered and washed with distilled water to remove residual soluble components. Precipitation pH was considered the primary variable influencing compositional distribution.

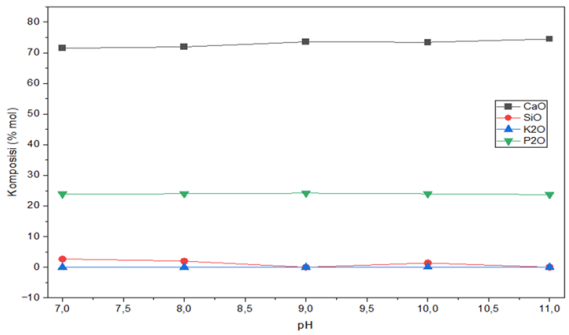
2.2.4. Calcination

The washed precipitates were dried prior to thermal treatment. Calcination was conducted at 600, 700, 800, 900, and 1000 °C to investigate the influence of temperature on oxide stabilization and structural evolution. Each sample was maintained at the target temperature for a fixed duration before cooling to room temperature under ambient conditions. Calcination temperature was treated as the second independent variable in this study.

3. Results and Discussion

This section presents and discusses the compositional behavior and yield characteristics of the synthesized $CaO–SiO_2–K_2O–P_2O_5$ composite as influenced by precipitation pH and calcination temperature. The oxide compositions determined by XRF are analyzed to evaluate how chemical conditions during precipitation and subsequent thermal treatment affect phase stabilization and oxide redistribution within the multicomponent system. In addition, yield data are examined to assess mass retention and thermal transformation behavior under varying synthesis parameters. The discussion integrates experimental observations with previously reported findings to provide a comprehensive interpretation of compositional evolution and process efficiency.

3.1. Effect of pH on Composition



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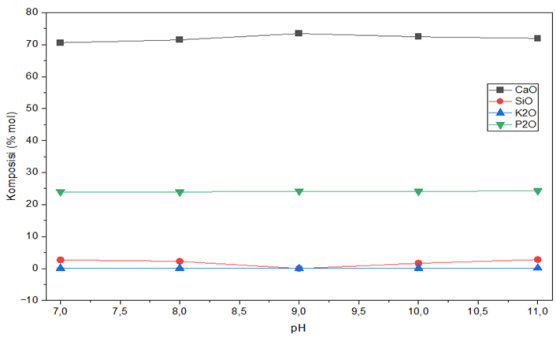

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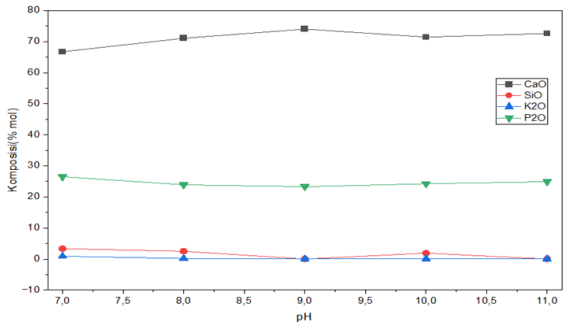

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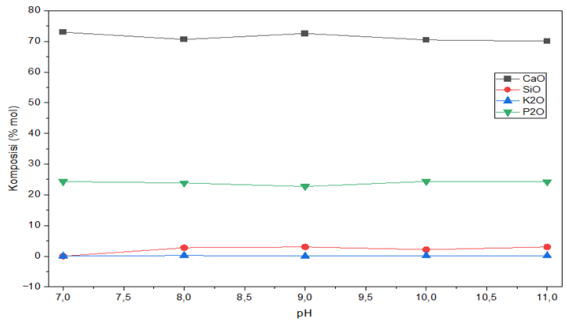

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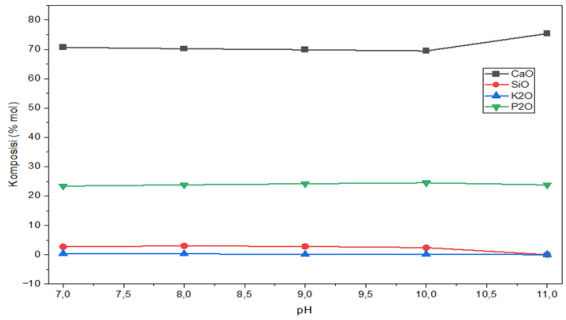
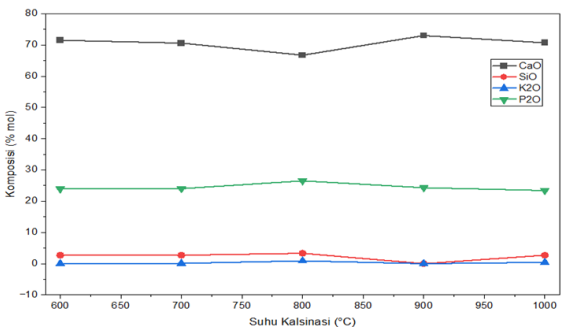


(e)

Fig. 1. Graph showing the effect of pH on the composition of CaO–SiO_2 –K_2 O–P_2 O_5 at calcination temperatures of (a) 600°C; (b) 700°C; (c) 800°C; (d) 900°C; and (e) 1000°C.

Based on Figure 1, variations in pH influence the composition of the CaO–SiO_2 –K_2 O–P_2 O_5 compounds at each calcination temperature. In general, increasing the pH from 7 to 11 enhances the amount of precipitated CaO, particularly within the neutral to slightly alkaline range (pH 7–8); however, at higher pH values (pH 8–9), the CaO content tends to stabilize, indicating reduced solubility and a near-saturation precipitation condition [12]. This behavior is associated with the increased availability of OH^- ions, which promote the deprotonation and decomposition of calcium precursors, thereby facilitating CaO formation. Conversely, the SiO_2 content decreases under highly alkaline conditions due to the high solubility and stability of silicate species in solution, causing more silica to remain in the filtrate rather than accumulate in the precipitate, as also reported by [13]. The measured K_2 O content remains relatively low, likely due to potassium loss during washing and filtration processes that can reduce K_2 O content by approximately 50% [14] as well as the volatility and migration of alkali ions during heat treatment [15], making the effect of pH on K_2 O less observable. Meanwhile, P_2 O_5 shows only minor fluctuations with pH changes, consistent with findings by [16] indicating that its solubility is relatively low and stable within the pH range of 7–11. Overall, across all calcination temperatures, pH variation exhibits a consistent trend in which CaO increases at pH 8–9, SiO_2 decreases under alkaline conditions, and K_2 O and P_2 O_5 remain relatively unchanged, demonstrating that alkaline conditions are the most favorable for the formation of calcium-based compounds in the CaO–SiO_2 –K_2 O–P_2 O_5 system.

3.2. Effect of Calcination Temperature on Composition



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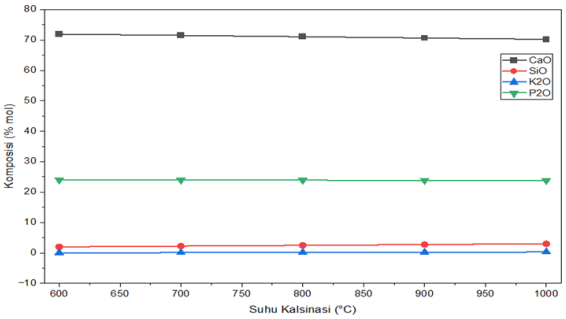

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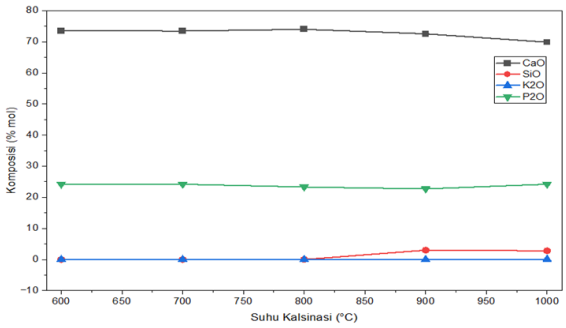

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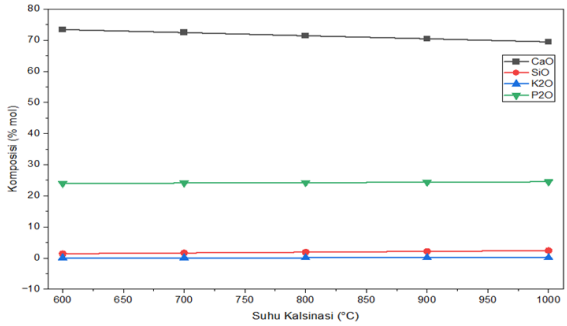

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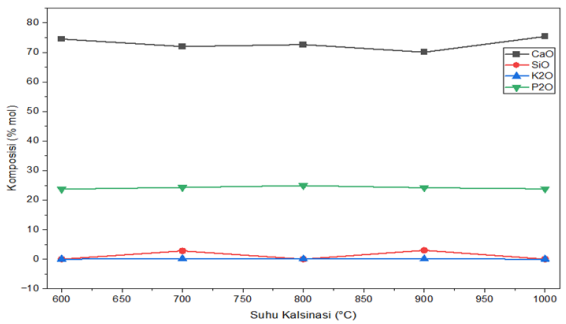

(e)

Fig. 2. Graph showing the effect of calcination temperature on the composition of CaO–SiO_2 –K_2 O–P_2 O_5 at pH values of (a) 7; (b) 8; (c) 9; (d) 10; and (e) 11.

Based on Figure 4, in the CaO–SiO_2 –K_2 O–P_2 O_5 system synthesized via the precipitation method, increasing the calcination temperature from 600 °C to 900–1000 °C can trigger solid–solid reactions and recrystallization processes that significantly redistribute oxide fractions, resulting in a final composition that deviates from the "standard bioactive" composition. The SiO_2 content tends to increase with rising calcination temperature, consistent with previous findings reporting higher SiO_2 fractions at 600 °C, 700 °C, 800 °C, 900 °C, and 1000 °C, which are attributed to the removal of impurities at elevated temperatures, thereby increasing the relative dominance of SiO_2 in the material [17]. The P_2 O_5 composition shows an increasing trend within the temperature range of 600–800 °C, followed by a decrease at 900–1000 °C, in agreement with [5], who reported a similar pattern of increasing P_2 O_5 content at 600–900 °C and subsequent reduction at higher temperatures. This trend suggests that phosphate species are relatively stable and well incorporated into the material matrix at intermediate temperatures, whereas at higher calcination temperatures, the reduction in P_2 O_5 fraction may be associated with phosphate volatility or phase reorganization during recrystallization, as P_2 O_5 release from molten or solid phases generally occurs at temperatures around 900 °C, which are higher than typical alkali volatilization thresholds. Meanwhile, although K_2 O may initially be present in relatively high amounts, its contribution in the final phase tends to decrease due to its higher volatility and the migration of alkali ions during heat treatment, thereby limiting its role in the final composition. In contrast, the CaO composition in this study decreases with increasing calcination temperature; however, this finding differs from previous studies reporting an increase in CaO content at higher calcination temperatures [18]. The discrepancy may be explained by the increasing SiO_2 content observed in this study, while P_2 O_5 and K_2 O remain relatively constant, leading to a proportional decrease in the CaO fraction in the overall composition.

3.3. Effect of pH on Yield

DOI:10.4186/ijeise.upnjatim.ac.id/E-ISSN: 2721-8775

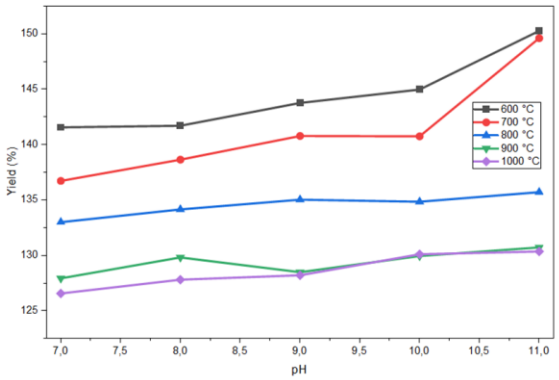

Fig. 3. Graph showing the effect of pH on yield (%)

Based on Figure 3, the obtained yield exceeds 100%, which can be attributed to the addition of 20% KOH solution during the experiment to adjust the pH. Increasing the pH from 7 to 11 enhances the yield of the $CaO–SiO_2 –K_2 O–P_2 O_5$ composite across the entire calcination temperature range, although with varying sensitivity depending on temperature, indicating a shift in the yield-controlling mechanism. At low to intermediate temperatures (600–700 °C), the sharp increase in yield under alkaline conditions suggests that chemical precipitation remains the dominant process, where enhanced PO_4^{3-} speciation and greater Ca^{2+} availability accelerate nucleation and growth of calcium–phosphate phases, resulting in higher solid mass formation. In addition, in this multicomponent system, alkaline conditions increase silicate solubility, promoting Si co-precipitation in amorphous or semi-crystalline phases and contributing to mass retention. However, at higher calcination temperatures (800–1000 °C), the influence of pH on yield becomes more moderate due to the dominance of thermal processes such as sintering and structural reorganization, as well as the role of $K_2 O$ as a flux that enhances densification and reduces product sensitivity to initial chemical conditions. Overall, these results indicate that alkaline pH conditions (9–11) represent the optimum range, balancing initial chemical reactivity and thermal stability, thereby maximizing Ca–P phase formation and silicate retention without causing yield reduction, as reported under extreme pH conditions in previous studies [19]

3.4. Effect of Calcination Temperature on Yield

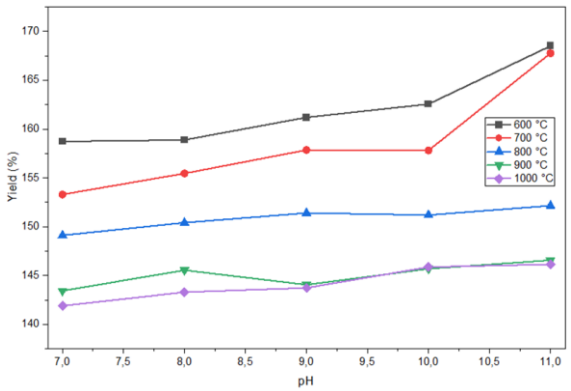

Fig. 4. Graph showing the effect of calcination temperature (°C) on yield (%).

Based on Figure 4, the obtained yield exceeds 100%, which can be attributed to the addition of 20% KOH solution during the experiment to adjust the pH. Increasing the calcination temperature shows an inverse relationship with the yield of the $CaO–SiO_2 –K_2 O–P_2 O_5$ composite, where relatively high yield values are observed at 600–700 °C, followed by a significant decrease in the 700–800 °C range, and then a tendency to stabilize at temperatures ≥900 °C. This pattern indicates a shift in the dominant mechanism during calcination; at intermediate temperatures, the most intense thermal transformations occur, including the release of bound water, loss of volatile fractions, and degradation of organic and carbonate components, all of which directly contribute to the reduction in residual mass. At higher temperatures, the relatively stable yield suggests that most devolatilization and decomposition processes have already taken place extensively, so further temperature increases primarily promote structural reorganization, densification, and solid-phase maturation rather than additional mass loss. These findings are consistent with the report by [20], who observed a decrease in residual mass from 47.59% at 600 °C to 41.84% at 1000 °C due to dehydration, decarbonation, and organic degradation during gradual heating. Overall, these results confirm that calcination temperature is a critical parameter that must be optimized to balance final structural stability and yield efficiency, in accordance with the thermal transformation characteristics of multicomponent material systems.

4. Conclusions

DOI:10.4186/ijeise.upnjatim.ac.id/E-ISSN: 2721-8775

Based on the research findings, it can be concluded that both variables play a significant role in determining the composition and yield of the material. Alkaline conditions (pH 9–11) are the most effective in increasing the CaO fraction and yield by accelerating Ca–P phase formation due to reduced calcium solubility and enhanced phosphate formation, while SiO_2 tends to decrease at high pH and K_2O and P_2O_5 remain relatively unchanged. Increasing the calcination temperature induces phase transformations and structural reorganization that alter oxide proportions, with a relative increase in SiO_2 and a decrease in K_2O and some of P_2O_5 due to volatilization, as well as a reduction in yield caused by dehydration and decarbonation until thermal stability is achieved. Although variations in pH and temperature result in noticeable compositional changes, the final composition does not yet meet bioactive standards for biomedical applications; however, it approaches the characteristics of composites used in the agricultural sector. Therefore, the synthesized material shows greater potential for application as a fertilizer or soil ameliorant, and further optimization of the synthesis method is required to obtain a composition that fulfills bioactive criteria.

Acknowledgement

The authors would like to express their sincere gratitude to the Department of Chemical Engineering, Universitas Pembangunan Nasional "Veteran" Jawa Timur, for providing laboratory facilities and technical support. Appreciation is also extended to PT. Dwigasindo Abadi for supplying carbide lime waste and to local farmers in Mojokerto, East Java, for providing rice husk used in this study. The authors further acknowledge the contributions of colleagues and laboratory assistants for their support during the research. This work did not receive any specific funding from public, commercial, or nor form profit funding agencies.

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