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GO-KNO₃ fertilizer: Slow release fertilizer innovation from coconut shell waste as a solution to Indonesian food security

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ABSTRACT

Background: As a country with a large agricultural sector, using fertilizers is an essential factor. Inorganic fertilizers such as KNO₃ are an option, but excessive use of fertilizers results in the accumulation of inorganic residues. The use of fertilizers that can release controlled nutrients is very necessary, one of which is by encapsulating with Graphene Oxide (GO). **Methods:** Coconut shell waste is used as the primary material for making GO which is synthesized by the Hummer method with variations in the mass of coconut shell graphite, the characterization of graphene oxide was Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), and Atomic Absorption Spectrophotometry (AAS). **Findings:** This research aims to synthesize GO and determine its characteristics as an encapsulation of KNO₃ fertilizer. **Conclusion:** The FTIR results obtained in this research detected O-H bonds, C-H bonds, and C = C bonds. In TEM characterization, thin morphology results were obtained, indicating an oxidation process in the formation of graphene oxide. The AAS showed that the release of KNO₃ from graphene oxide was maximum after 8 hours with a percentage of 93.8%. This fertilizer will be used to solve the problem of low plant absorption of macronutrients contained in fertilizers. **Novelty/Originality of this article:** Encapsulating KNO₃ fertilizer using GO to control nutrient release is a novel approach. This technique addresses the challenge of nutrient overuse and minimizes the environmental impact of inorganic fertilizers.

KEYWORDS: Graphene Oxide; agricultural sector; hummer method.

1. Introduction

Indonesia experiences a tropical climate characterized by two distinct monsoonal seasons, namely wet and dry. The average annual rainfall ranges from 1,780 to 3,175 mm, with certain areas receiving as much as 6,500 mm. The humidity is consistently high, typically between 70 and 80%. Situated on the edge of the Pacific Rim, Indonesia is home to numerous volcanoes. Its extensive and fertile soils make the country a key global producer of a wide variety of tropical agricultural products (Syuaib, 2016). Moreover, agriculture in Indonesia encompasses a range of activities, including rice cultivation, palm oil production, and fishing, which are critical for food security and the local economy (David & Ardiansyah 2017). Indonesia boasts a substantial land area of approximately 7.46 million square kilometers, which plays a crucial role in the robust growth of its agricultural sector (Hikmat et al., 2023). This vast expanse of land provides diverse climates and fertile soil, enabling

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the cultivation of a wide variety of crops and the raising of livestock. As a result, the agricultural sector has become a cornerstone of the Indonesian economy. In the year 2012, the Agricultural Gross Domestic Product (GDP) of Indonesia was reported at an impressive 327,549.7 billion rupiah. This figure reflects a consistent upward trend in agricultural productivity and output over the years. Notably, this growth trajectory has remained steady, except for a significant setback during the economic crisis of 1998, which saw a temporary decline in agricultural performance (Irham & Mulyo, 2016). Overall, the resilience of Indonesia's agricultural sector is evident in its capacity for annual growth and adaptation to changing economic conditions.

Fertilizers play a crucial role in boosting productivity within Indonesia's agricultural sector, making them essential for enhancing agricultural production, however the rapid release of large amounts of chemicals from inorganic fertilizers can lead to crop damage and environmental pollution (Krein et al, 2023). Chemical fertilizers were created to increase food production and solve the food crisis. Traditional commercial fertilizers are prone to wastage, resulting in low fertilizer utilization, and loss of resources (Paungfoo-Lonhienne et al., 2019). Conservative estimates suggest that 30-50% of agriculture is attributed to inorganic fertilizers. As modern agriculture increasingly relies on non-renewable fertilizer resources, associated minerals in the future are likely to yield lower quality at higher price (Cordell et al., 2009). Some of the nutrients in such non-renewable fertilizers are not absorbed by plants and therefore, leach into groundwater or surface water, causing eutrophication, and posing major risks to ecosystems (Schindler, 1974; Conley et al., 2009). The use of these fertilizers makes a significant impact, as it helps farmers maximize crop yields and ensure a sustainable food supply for the nation (Kurniawan et al., 2023). Therefore, using fertilizers is critical to increasing agricultural productivity because not all agricultural land in Indonesia has the same level of fertility and is suitable for all types of plants. Utilizing fertilizers in Indonesia is also enhancing, reaching 16 million tons or 2.4% each year (Khairani et al., 2022). Overall, farmers use inorganic fertilizers because of relatively low prices and the current limited production of organic fertilizers.

Potassium nitrate (KNO₃) fertilizer, which is recognized for its balanced supply of essential nutrients, including nitrogen (N), phosphorus (P), and potassium (K). The nitrogen component is crucial for promoting healthy foliage and overall plant vigor, while phosphorus is vital for root development and flowering. Additionally, potassium plays a significant role in various physiological processes, including water regulation and enzyme activation. The specific formulation of KNO3 fertilizer not only meets the nutritional requirements of various crops but also optimizes their yield potential (Abdulazeez et al., 2018; Mahajan et al., 2020). KNO₃ fertilizer is an inorganic product that enhances the availability of macronutrients, specifically nitrogen and potassium. It serves as a substitute for single fertilizers such as urea and potassium chloride (KCL), which can sometimes be difficult to find and quite expensive (Abdulazeez et al., 2018). The benefits of using KNO₃ fertilizer include its provision of the same essential nutrients as single fertilizers, its simplicity of use, and its efficiency in terms of transportation and storage. Additionally, nearly all types of this fertilizer dissolve in water, allowing for immediate absorption by plants, which promotes effective nutrient uptake (Dhillon et al., 2021). KNO₃ fertilizer is also characterized by a slow release of nutrients, which helps to minimize losses due to evaporation, leaching, and absorption by soil colloids. Furthermore, KNO3 can play a role in the encapsulation process in slow-release fertilizers (SRF). During encapsulation, hydroxyapatite fertilizer is coated with macronutrient compounds like KNO₃, which is intended to reduce nitrogen loss in the soil (Hamzah et al., 2019). However, excessive use of KNO₃ fertilizer can result in decreased soil quality and the soil cannot receive nutrients due to the accumulation of inorganic residues that are difficult to decompose naturally (Dhillon et al., 2021). Therefore, fertilizers must be designed to release their nutrients in alignment with the plants' nutrient absorption needs over time. By synchronizing nutrient release with the plants' growth stages, we can enhance nutrient uptake efficiency, minimize waste, and ultimately promote healthier and more vigorous plant growth.

Interest in the application of bio-based materials from sustainable and environmentally friendly sources is growing, coconut shells are one of the natural materials that can be used as composite materials (Darianto et al., 2019). Coconut shells can produce charcoal obtained from incomplete combustion, so that it can leave nutrients that are useful for the soil. If the combustion is perfect, the charcoal will turn into ash and remove carbon. Coconut shell charcoal can fertilize and moisturize the soil and increase land and plant productivity, cation exchange capacity, as well as organic C and rice yields. The charcoal contains nutrients such as sodium, magnesium, phosphorus, potassium, and others (Fitriatin et al., 2020). Generally, coconut shell charcoal is used for activated carbon as an absorber or adsorbent of impurities in water, but it is also possible to be used as a raw material in the manufacture of GO (Putri & Supardi, 2023). The provision of charcoal (biochar) to the soil can increase the population and activity of microbes and increase the provision of nutrients and habitat modification. In addition, the morphology of charcoal has pores that are very effective in storing and binding nutrients. These nutrients are released slowly according to plant needs (Fitriatin et al., 2020). These coconut shells serve as an excellent carbon source due to their composition, which includes significant amounts of organic compounds such as cellulose, lignin, and hemicellulose (Darianto et al., 2019). When processed, coconut shell charcoal emerges as a particularly valuable raw material for the production of graphite, attributed to its high carbon content and structure, which allows for efficient conversion. In addition to its applications in industrial processes, coconut shell charcoal possesses unique properties that enhance soil fertility. The incorporation of this charcoal into agricultural soils can lead to improved nutrient retention, increased microbial activity, and enhanced water retention. These benefits contribute to more robust crop growth and ultimately result in greater agricultural productivity and quality (Putri & Supardi, 2023). Thus, utilizing coconut shell charcoal not only aids in resource recycling but also plays a significant role in sustainable farming practices.

Modern agriculture's reliance on non-renewable fertilizers may result in lower quality and higher prices in the future. Many nutrients in these fertilizers are not absorbed by plants, causing them to leach into water sources and contribute to eutrophication, which harms ecosystems. To enhance fertilizer quality and protect the environment, research is increasingly focused on developing technologies that deliver nutrients in a slow or controlled manner (Zhang et al., 2014). Slow Release Fertilizer (SRF) is a fertilizer that can increase the efficiency of nutrient absorption from fertilizers (Hamzah et al., 2019). Currently, there are many prevalent strategies for manufacturing sustained-release fertilizers. One approach involves the preparation of slow-release fertilizers through traditional chemical synthesis to form complexes or chelates (Duan et al., 2023). In addition, slow-release fertilizers can be formed by loading nutrients into porous or layered inorganic materials, such as kaolinite, montmorillonite, and zeolite. For example, montmorillonite and glauconite, when used as raw materials and mechanically activated, can be mixed with urea to synthesize slow-release fertilizers (Rudmin et al., 2020; Yan et al., 2021). Moreover, another strategy includes coating the surface of nutrient sources with inorganic minerals or organic polymers, thereby developing slow-release fertilizers with functional biodegradable coatings (Sim et al., 2021; Ye et al., 2020). Furthermore, nutrients can be efficiently loaded using physicochemical interactions by preparing hydrogel-type sustained-release fertilizers, which have the ability to retain many times their own mass in water (Kenawy et al., 2020; Liu et al., 2022). This type of high-efficiency fertilizer reduces nutrient loss and improves utilization efficiency through the slow release of nutrient elements by diffusion, dissolution or other mechanisms (Lu et al., 2022).

The advantages of using SRF fertilizers are reducing environmental pollution and saving fertilizer consumption (Azizah et al., 2021). The release of nutrients from SRF fertilizers can be regulated so that fertilizer nutrients do not dissolve quickly in water, which can be done by encapsulating protection from semipermeable materials or permeable porous materials (Noor et al., 2022). Encapsulation is an innovative technique employed to create slow release fertilizers (SRF) by enveloping conventional fertilizer granules with specific materials that regulate the rate at which the granules dissolve. This

process underscores the significant role that the choice of coating material plays in determining how quickly or slowly the nutrients are made available to plants. Current approaches to achieve slow release of fertilizers usually involve coating measures. However, the application of non-biodegradable petrochemical polymer coatings has a pronounced ecological impact, leading to higher costs and pollution during the production and application phases (Katsumi et al., 2021; Weng et al., 2023). Given these challenges, there is an urgent need for innovative and efficient strategies that are not only cost-effective and environmentally friendly but also proficient in effectively encapsulating KNO₃ fertilizers and achieving their long-term release. Micro-/nanomaterials are widely used in agriculture due to their distinctive physicochemical properties (Hassanisaadi et al., 2023; Ran et al., 2023). Microencapsulation technology, a form of micropackaging for solids, liquids, and gases, has advanced rapidly since the 20th century (Petrulis & Petrulyte, 2019). Considering the relatively low mechanical performance requirements for shell materials, microencapsulation technology has considerable research potential and practical value in various fields such as medicine (De Cock et al., 2010), food (Calderón-Oliver & Ponce-Alquicira, 2022), pesticides (Li et al., 2023), and cosmetics (Pratiwi et al., 2022).

Among the various materials explored for this purpose, GO has emerged as a promising candidate. Research by Kabiri et al. (2017) demonstrates that GO serves as an effective carrier for essential nutrients. Its unique properties allow for the gradual release of micronutrients, particularly zinc (Zn) and copper (Cu), which are crucial for plant health and development. The use of GO not only facilitates a controlled and sustained nutrient delivery system but also enhances the overall efficiency of nutrient uptake by plants, potentially reducing the need for frequent fertilization and minimizing environmental impacts associated with nutrient runoff. This advancement illustrates the promising future of fertilizer technology, enhancing agricultural productivity while promoting sustainable practices.

Graphene oxide (GO) is a distinct material known for its remarkable chemical properties, adsorption capabilities, and biocompatibility. It possesses a two-dimensional (2D) structure, a high surface area (2600 m²/g), is economically produced, and can interact directly with a variety of biomolecules and nanoparticles. Furthermore, its structure is enhanced with various functional groups, such as epoxy (-CH(O)CH-), hydroxyl (-OH), carboxyl (-COOH), and ester (-COO-) groups, which make it an ideal platform for functionalization. Additionally, Guo et al. (2009) presented a simple method for producing high-quality graphene nanosheets on a large scale through the electrochemical reduction of exfoliated graphite oxide precursor at cathodic potentials.

Graphene exhibits exceptional properties, including a high specific surface area, mechanical strength, chemical and thermal stability, low toxicity, and a surface chemistry that can be easily modified (Ghoshal et al., 2024). In agriculture, GO-based nanomaterials have been demonstrated to enhance the efficiency of agrochemicals by enabling targeted delivery and slow-release action, as well as improving plant growth and development in the soil (Perreault et al., 2015). Research by Shekari et al. (2017) highlighted the benefits of GO nanoparticles (NPs) in enhancing agricultural crop productivity, with soil-based GO treatments significantly improving the soil's physicochemical properties. Furthermore, Lalwani et al. (2014) showed that lignin peroxidase, a ligninolytic enzyme from white-rot fungi, effectively degrades GO in the soil, converting it into a completely safe product, thus preventing secondary environmental pollution. A study on Silybum marianum demonstrated that the application of GO increased chlorophyll content and relative water content (RWC), promoting plant growth potential and improving crop yield (Yang et al., 2022).

The fabrication and modification process for GO is relatively easy and inexpensive, allowing versatility in its use across a wide range of industries. In addition, its structure provides two external surfaces, effectively doubling the active sites available for adsorption compared to materials that have only one surface. Most importantly, GO does not contain toxic metal particles, making it a safer choice for environmental and health applications. Because of its unique morphological structure and related properties, graphene has been

reported to be an effective carrier for various chemical compounds, thus holding potential opportunities for developing new controlled release delivery systems (Liu et al., 2008; Yang et al., 2009, 2011; Zu & Han, 2009). For example, Yang et al. developed a simple, effective, and scalable method to chemically deposit Fe3O4 nanoparticles onto GO. This hybrid can be loaded with the anti-cancer drug DXR with a high loading capacity (Yang et al., 2009). However, besides those medical applications, little research has been done to explore graphene-based slow- and controlled release systems for agricultural applications such as fertilizers, pesticides and so forth.

In the research conducted by Zhang et al. (2014), SRF fertilizer developed by encapsulating KNO₃ pellets using GO film at 90°C for 6 hours in air. The results showed that with the help of potassium ions, the separated GO sheets not only coalesced to form a shell around KNO₃ but also reduced to re-GO sheets during heat treatment. The resulting re-GO coated KNO₃ pellets exhibit slow release behavior. Due to the unique characteristics of graphene, this newly developed method can be used to produce fertilizers in a controlled-release manner, deliver nutrients to crops, increase crop productivity, and minimize nutrient losses, especially when graphene/GO can be produced on a large scale with environmentally friendly methods and relatively low costs.

There are several methods utilized in the synthesis of GO, each with distinct advantages and disadvantages. Notable examples include the Staudenmaier and Brodie methods, both of which employ potassium chlorate in conjunction with various acids. While effective, these methods have significant drawbacks; they produce toxic by products, particularly chlorine dioxide (ClO_2) gas, which poses serious health and safety risks (Lu et al., 2013). The Brodie method, despite its efficiency in producing GO that readily disperses in alkaline solutions, results in products characterized by an imperfect structure. This structural flaw can limit the potential applications of the synthesized GO. On the other hand, the Staudenmaier method is fraught with its own challenges, chiefly a lengthy synthesis process and a notable risk of explosion during production (Cardoso et al., 2019). Among the various synthesis methods, the Hummer method stands out as one of the most effective. This approach is favored due to its environmentally friendly characteristics as it does not release harmful ClO₂ gas and produces products with a much higher oxidation state compared to those produced by the Staudenmaier method. The increased oxidation state enhances the functional properties of GO, making it more suitable for various applications (Umar & Burhendi, 2022; Cardoso et al., 2019). The materials required for the Hummer method are also relatively easy to procure, making it a practical choice for researchers and manufacturers alike. As a result, this method is widely employed in the synthesis of GO (Yu et al., 2016). In light of the challenges associated with traditional synthesis methods, innovative research has been directed towards utilizing GO derived from coconut shell waste for agricultural applications. Specifically, this approach aims to develop a slowrelease fertilizer (SRF) that leverages the unique properties of GO.

The adoption of GO-KNO₃ fertilizer presents significant socio-economic and policy implications, especially in improving agricultural productivity and farmers' welfare. In a study conducted by Putri et al. (2024), it was shown that GO-KNO₃ fertilizer has the potential to increase crop yields, similar to the positive effect observed with NPK fertilizer, which increased rice yield by 0.198%. Increased yields can lead to higher incomes for farmers, addressing the current challenges posed by rising production costs due to rising global fertilizer prices, thereby improving food security, reducing risks associated with food price inflation and poverty (Mulyono et al., 2023).

In the present study, addresses a critical gap in the field of slow-release fertilizers (SRFs) by leveraging the potential of GO synthesized from renewable coconut shell waste to encapsulate KNO₃ fertilizer. While traditional SRFs often face challenges related to inconsistent nutrient release rates, high production costs, and environmental concerns due to synthetic materials, this research presents a sustainable and efficient alternative. The novel approach of using GO, characterized by its thin morphology and functional groups such as O-H, C-H, and C=C bonds confirmed through FTIR, ensures controlled nutrient release as demonstrated by AAS analysis. By addressing the inefficiencies of conventional

inorganic fertilizers, such as poor nutrient absorption and environmental residue accumulation, this study not only improves fertilizer efficiency but also contributes to sustainable agricultural practices in Indonesia. The findings advance the current knowledge by integrating advanced material science with practical agricultural applications, offering a scalable solution to enhance food security and reduce the ecological footprint of fertilizer usage.

2. Methods

2.1 Materials

In this research, a variety of materials were utilized to conduct experiments. The primary chemical reagents included 98% sulfuric acid (H₂SO₄), sodium nitrate (NaNO₃), and hydrogen peroxide at a concentration of 30% (H₂O₂). Additionally, potassium nitrate (KNO₃) and a 10% hydrochloric acid solution (HCl) were employed. Distilled water was used throughout the procedures to ensure purity and prevent contamination. Furthermore, commercial coconut shell waste was incorporated as a key component in the study. To facilitate the research, a range of laboratory instruments and equipment were employed. The glassware consisted of beakers, flasks, and pipettes, all essential for mixing and measuring reagents accurately. An analytical balance was used for precise measurements of solid materials. Temperature control was managed using a thermometer, an ice bath for cooling applications, and a hot plate equipped with a magnetic stirrer for maintaining homogenous mixtures during heating. Advanced techniques were applied for material characterization using several sophisticated instruments. A sonicator was utilized to enhance the reaction kinetics and improve the dispersion of particles in solution. A centrifuge was employed to separate components based on density, while an oven was used for drying samples as needed.

To determine the pH levels of solutions during various stages of experimentation, a pH meter was used, ensuring that all reactions were conducted under optimal conditions. Furthermore, an FTIR spectrometer (PerkinElmer Spectrum Two LiTa) was utilized for identifying functional groups and molecular structures. The presence and concentration of different elements were analyzed using Atomic Absorption Spectroscopy (AAS) with a Perkin Elmer PinAAcle 900T. For assessing the morphology and particle size of samples, Transmission Electron Microscopy (TEM) with a Thermo Fisher Type Axia HV was employed. Lastly, X-ray Diffraction (XRD) analysis was conducted with a Malvern Panalytical Tpe Empyrean to investigate the crystallographic structure of the materials used. This comprehensive approach ensured thorough investigation and reliable results throughout the study.

2.2 Synthesis of graphite from coconut shell waste

Graphite was synthesized from coconut shells through a method known as the carbonation process, which involves the thermal decomposition of raw organic material. For this procedure, a precise sample of 100 grams of dried coconut shells was first selected to ensure consistency in the experiment. The dried coconut shells were then placed into a heating furnace specifically designed for high-temperature applications. The furnace was set to a temperature of 1000°C, and the shells underwent a controlled burning process for a duration of one hour. This intense heat caused the organic material to decompose, leading to the formation of charcoal. Once the burning process was completed, the resulting coconut shell charcoal was carefully removed from the furnace and allowed to cool down to room temperature. After cooling, the charcoal was weighed to determine its yield from the initial 100 grams of coconut shells. The charcoal was then ground into a fine powder to increase its surface area for any further applications. Finally, the powdered charcoal was reweighed to assess any changes in mass that occurred during the carbonation process. This entire

procedure highlights the transformation of coconut shells into graphite through careful thermal processing.

2.3 Synthesis of graphite oxide

Graphite oxide was synthesized using the Hummer method by dissolving graphite with variations of 0.5 grams, 1 gram, and 1.5 grams in 98% $\rm H_2SO_4$ solution and stirring in an ice bath at 0-5°C for 2 hours. In addition, NaNO₃ was added and stirred using a magnetic stirrer for 2 hours. Then the sample was stirred for 24 hours at room temperature. After distilled water was added slowly, it was stirred for 1 hour. Add distilled water and 30% $\rm H_2O_2$, subsequently, centrifuge until the entire sample settles. Finally, the sample was dried in an oven at 80°C for 12 hours. Graphite oxide was synthesized following the Hummers method, a well-established technique in materials science. The process began by dissolving varying amounts of graphite—specifically 0.5 grams, 1 gram, and 1.5 grams—in a concentrated 98% sulfuric acid ($\rm H_2SO_4$) solution. This mixture was carefully stirred in an ice bath maintained at a temperature range of 0-5°C for a duration of 2 hours to ensure controlled oxidation of the graphite.

Following this initial stirring period, sodium nitrate (NaNO₃) was introduced into the solution. The mixture was subjected to continuous stirring using a magnetic stirrer for an additional 2 hours. This step was crucial as NaNO₃ acts as an oxidizing agent, significantly enhancing the intercalation of the graphite layers. After completing the second stirring phase, the sample stood and was stirred at room temperature for 24 hours. This prolonged stirring facilitated further oxidation of the graphite. Next, distilled water was added gradually to the mixture, a process done slowly to manage the exothermic reaction that occurs between the acid and water. The combined mixture was then stirred for another hour to ensure thorough mixing and complete reaction of the components.

After that, an appropriate quantity of 30% hydrogen peroxide (H_2O_2) was incorporated into the solution. This step serves to neutralize the excess sulfuric acid and further assist in the oxidation of the graphite. The reaction was allowed to proceed, after which the solution was centrifuged to separate the synthesized graphite oxide from the liquid phase, ensuring that the entire sample settled properly. Finally, the collected graphite oxide was dried in an oven set at 80°C for 12 hours. This drying process removed any remaining moisture and allowed for the preparation of the graphite oxide for further characterization or applications.

2.4 Synthesis of GO

Graphite Oxide was carefully measured and placed into a vial, then an equal volume of distilled water was added to achieve a 1:1 ratio of Graphite Oxide to water. The mixture was subjected to sonication for a duration of 90 minutes to ensure thorough dispersion of the Graphite Oxide particles in the liquid medium. Following sonication, the sample was centrifuged until all solid particles settled at the bottom of the vial, confirming the separation of the liquid and solid phases. To finalize the process, the supernatant was removed, and the remaining solid was placed in an oven set to a temperature of 80°C. The sample was dried for a period of 12 hours to ensure complete removal of any residual moisture, resulting in a well-prepared Graphite Oxide product.

2.5 Preparation of GO coating

GO was dissolved in a 20 mL solution of distilled water. This process was conducted under sonication for a duration of 2 hours, which ensured complete dispersion of the GO particles in the solution. Following sonication, the GO was subjected to a filtration process to separate any undissolved materials. After filtration, the GO was allowed to dry naturally at room temperature, ensuring it retained its structural properties. Subsequently, KNO₃ fertilizer was carefully mixed into the dried GO. To facilitate the binding and uniform

distribution of the fertilizer, a small amount of water was added to the mixture. The resulting composite was then placed in a preheated oven at a temperature of 90°C for a continuous period of 6 hours. This thermal treatment aimed to enhance the interaction between the GO and potassium nitrate. After the heating period, the sample was removed from the oven and allowed to cool gradually back to room temperature, completing the preparation process.

2.6 Release testing of graphene coated KNO3 fertilizer

Fertilizer samples were measured and put into sterile bottles containing 100 mL of high-purity distilled water to ensure that no contaminants affected the results. The bottles were then sealed and stored in an incubator with a controlled temperature of 25°C, which was considered optimal for the experimental conditions. At predetermined time intervals of 0.5 hours and 1 hour, 2 mL of distilled water solution was taken from the bottle using a sterile pipette. This solution was then diluted by adding 2 mL of distilled water in a separate container to ensure consistency of analysis. After dilution, the mixture was injected back into the original bottle to maintain the volume and concentration of the initial solution. Analysis of the potassium content of the samples was performed using Atomic Absorption Spectrophotometry (AAS), which works by measuring the absorption of light at specific wavelengths, providing accurate quantification of the element.

In this study, the fertilizer release process was tested for a specific duration according to the rate at which potassium ions were completely absorbed by the water. In addition, environmental precautions have been implemented, such as the use of minimal amounts of chemicals and the disposal of waste with environmentally friendly procedures.

2.7 Characterization of GO

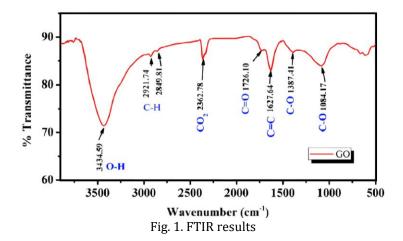
The characterization of GO conducted in this research involved a series of comprehensive analytical techniques to elucidate its properties and functionality. Fourier Transform Infrared Spectroscopy (FTIR) was utilized to identify the various functional groups present in the GO sample. This technique enables the detection of specific molecular vibrations, providing insight into the chemical structure and bonding types within the material. Next, X-Ray Diffraction (XRD) analysis was performed to assess the degree of crystallinity and to determine the crystalline phases present in the GO and graphene itself. By measuring the angles and intensities of the diffracted X-rays, we were able to derive information about the arrangement of atoms in the material and the extent of its crystalline order. The Transmission Electron Microscope (TEM) was employed to visualize and characterize the morphological features of the GO-coated KNO₃ fertilizer sample. This technique provided detailed images that reflect the two-dimensional structure of the GO and its distribution on the fertilizer, allowing for an assessment of the coating uniformity and thickness. Atomic Absorption Spectrophotometry (AAS) was used to quantify the potassium content in the fertilizer coated with GO. This analytical method relies on the absorption of light by free atoms, providing accurate measurements of potassium levels, which is crucial for evaluating the efficacy of the fertilizer in agricultural applications. Through these advanced characterization methods, we gained a comprehensive understanding of the properties of GO and its potential applications in enhancing the performance of KNO₃ fertilizer.

3. Results and Discussion

3.1 FTIR results

Fourier Transform Infrared (FTIR) spectroscopy was employed to comprehensively identify the various functional groups present in the samples under investigation. The analysis revealed several distinct oxygen-containing configurations within the molecular

structure. In Figure 1, the spectroscopic data indicated the presence of epoxide vibrational modes, characterized by C-O-C stretches observed in the wavenumber range of 1230 to $1320~\rm cm^{-1}$. This suggests the existence of cyclic ether structures within the samples. Additionally, peaks corresponding to $\rm sp^2$ hybridized carbon-carbon double bonds (C=C) were detected between 1500 and 1600 cm⁻¹, highlighting the presence of alkenes or aromatic compounds within the samples. Furthermore, the analysis identified carboxyl groups (COOH), with significant absorption bands appearing in the region of 1650 to 1750 cm⁻¹, indicative of the carbonyl (C=O) stretching vibrations. The C-OH vibrations associated with carboxylic acid and alcohol functionalities were discernible at approximately 3530 cm⁻¹ and 1080 cm⁻¹ respectively respectively as per the research conducted by Yu et al. (2016).



In addition, ketonic species were detected, with their characteristic C=0 stretching vibrations found between 1600 and 1650 cm⁻¹ as well as in the range of 1750 to 1850 cm⁻¹, further supporting the presence of carbonyl functions in the samples. Lastly, the FTIR spectrum indicated hydroxyl groups, specifically phenolic structures, identified by their C-OH stretching vibrations observed between 3050 and 3800 cm⁻¹, alongside additional bands at 1070 cm⁻¹. Notably, these vibrations encompass contributions from both the carboxyl groups and water (H_2O) present within the samples, underscoring the complex interplay of functional groups within the analyzed materials.

3.2 XRD results

X-ray diffraction (XRD) techniques play a crucial role in uncovering the unique material properties of nanostructured thin films and bulk crystal samples. These methods provide a fascinating glimpse into the intricate structures and behaviors of materials at the nanoscale, allowing researchers to explore and understand their characteristics in depth (Pandey et al., 2021). XRD characterization is to determine the crystal phase contained in GO. Figure 2 is a display of the XRD results of GO. The image provided presents the X-ray diffraction (XRD) patterns for both graphite and GO, offering insights into their respective crystal structures. In the case of graphite, several distinct peaks are observed, specifically at the positions indexed as (101) at 50.68°, (004) at 54.62°, (110) at 59.84°, and (112) at 71.56°. These peaks confirm the hexagonal arrangement of carbon atoms characteristic of graphite, indicating its well-defined and orderly crystalline structure. One of the most notable features of the graphite pattern is the pronounced (002) peak observed at 26.56°. This peak is critical as it reveals an interplanar spacing, denoted as 002, of 0.334 nm. This relatively small interplanar distance is indicative of graphite's stability and high degree of crystallinity, which is a result of strong van der Waals forces acting between the graphene layers, allowing them to maintain their orientation effectively.

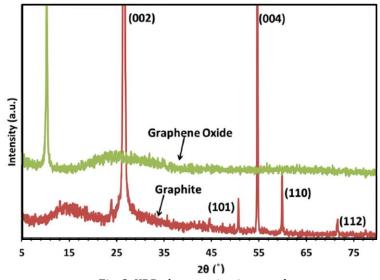


Fig. 2. XRD characterization results

In contrast, the XRD pattern of GO reveals a significantly different characteristic. It exhibits a prominent and sharp peak centered at 10.24°, which corresponds to an interplanar distance of 0.80 nm. This notable increase in interplanar spacing compared to graphite is attributed to the introduction of various oxygen-containing functional groups, such as hydroxyl, epoxide, and carboxyl groups. Additionally, the presence of some structural defects in GO contributes to the alteration of its crystalline order, thereby resulting in this elevated d-spacing. The structural changes in GO highlight the impact of functionalization on the material's properties, making it distinct from its graphite precursor.

3.3 TEM results

Transmission electron microscopy (TEM) is a powerful imaging technique that uses a beam of electrons transmitted through a thin sample, allowing for detailed visualization of its interior. This method is widely employed in nanomedical research, as it can reveal intricate relationships between nanoparticulates and cell or tissue components, thanks to its high resolution. The short wavelength of the electron beam—about 100,000 times shorter than that of visible light—enables TEM to achieve sub-nanometer resolution, approximately 0.2 nm in conventional setups (Malatesta, 2021). The TEM test aimed to determine the 2D projection of the KNO3 fertilizer sample coated with GO. Figure 3 shows the TEM result of GO sample. TEM image demonstrates the presence of substantial GO sheets, each measuring a minimum of 10 nanometers. The image prominently displays a ring pattern alongside a hexagonal symmetry point pattern, definitively highlighting the structural characteristics of the GO. The ring patterns unmistakably indicate multiple orientations of the GO sheets, which arise from the inherent wrinkling and folding of the layers, as well as the overlapping of different GO sheets. This complexity is a clear reflection of the dynamic interactions occurring during the material's synthesis.

Moreover, the distinct dot pattern represents major single crystal domains within the material, showcasing sp-hybridized carbon arranged in a hexagonal lattice formation. This specific arrangement is indicative of graphene's unique structural properties and underscores its substantial potential for diverse applications. These findings convincingly confirm that the GO produced using potassium nitrate (KNO₃) fertilizer results in well-reduced GO sheets. These sheets can exist as few-layer or multi-layer configurations, directly affecting their physical and chemical properties, making them suitable for significant applications in advanced materials and nanotechnology.

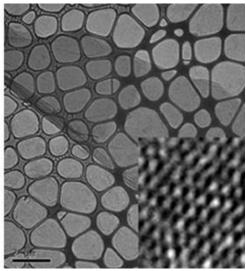


Fig. 3. TEM results

3.4 Release of KNO₃ fertilizer coated by GO

In Figure 4, Ck+ indicates the concentration of potassium ions in the elutrient. The data indicates that the release rate of potassium varies significantly across different time stages when utilizing GO sheets as a coating to postpone the entire release. This variation effectively prolongs the overall potassium release process. In the initial phase, spanning from 0 to 7 hours, the potassium release rate is notably slow when compared to subsequent stages.

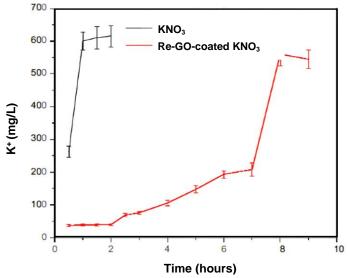


Fig. 4. AAS test results of KNO₃ fertilizer release coated with GO

This test shows that the GO coating can provide good controlled release, in contrast to the KNO_3 fertilizer without coating carried out by Zhang et al. (2014). During this period, only approximately 34.5% of potassium ions are released into the surrounding water. This slow release can primarily be attributed to the limited diffusion of water molecules through the GO shell. The water must penetrate the coating to reach the KNO_3 core, which is essential for establishing a release channel through which the encapsulated potassium ions can exit. Transitioning to the next phase, between 7 to 8 hours, there is a marked increase in the release rate. During this critical timeframe, around 93.8% of the total potassium is released from the fertilizer, the burst release of potassium ions takes place in the stage from 7 to 8 h same as the research of Cheng et al. (2024) which is 94.45% of the potassium ions were released from the fertilizers. This sudden spike can be explained by the earlier formation of

effective pathways for ion release, allowing for a rapid outflow of potassium ions once the initial diffusion barrier is overcome. Therefore, the use of GO sheets significantly influences the timing and efficiency of potassium ion release from the coated fertilizer.

4. Conclusions

GO was synthesized from coconut shell waste using the Hummers method. The process involved the oxidation of graphite with KMnO₄ in a solution of NaNO₃ and H₂SO₄, resulting in the formation of GO with oxygen-containing functional groups like epoxide, carboxyl, and hydroxyl, confirmed through FTIR analysis. XRD revealed a characteristic peak at 10.24° with an interlayer spacing of 0.80 nm, indicating successful oxidation. TEM images showed graphene sheets with varied layers. The GO's potassium release profile demonstrated an initial slow release (34.5% in 7 hours), followed by a significant increase (93.8% at 8 hours), highlighting its potential for controlled nutrient release in NPK fertilizers. These results indicate that with the help of potassium ions, the separated GO sheets not only coalesce to form a shell on KNO₃, but are also reduced to re-GO sheets during heat treatment. The prepared re-GO coated KNO₃ pellets exhibited slow release behavior. This effectiveness is due to the ability of GO to slow down the diffusion rate of water through its protective layer, thus allowing for a more controlled and sustained release of nutrients. In this way, fertilizers can deliver nutrients according to crop needs, improve nutrient absorption efficiency, and reduce waste and harmful residue accumulation in the soil. This innovation improves fertilization efficiency by means of controlled release, delivering nutrients to crops, increasing crop productivity, and minimizing nutrient losses, especially when graphene/GO can be produced on a large scale with environmentally friendly methods, relatively low costs and increase agricultural productivity but also support sustainable farming practices, making it a promising solution to food security challenges in Indonesia and internationally.

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Author Contribution

Conceptualization, A.A.M and D.M.N.; Methodology, R.A.Q.; Software, A.A.M.; Validation, A.A.M., D.M.N, and R.A.Q.; Formal Analysis, D.M.N.; Investigation, R.A.Q.; Resources, D.M.N.; Data Curation, D.M.N; Writing – Original Draft Preparation, A.A.M.; Writing – Review & Editing, D.M.N. and H.S.; Visualization, D.M.N.; Supervision, H.S.; Project Administration, A.A.M.; and Funding Acquisition, R.A.Q.

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Primary data must be requested to uphold confidentiality and adhere to ethical standards.

Conflicts of Interest

The authors declare no conflict of interest.

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