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Synthesis and Characterization of Silica-Potassium-Phosphate Solid Fertilizer Derived from Sugarcane Bagasse Ash

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A B S T R A C T

This research aimed to synthesise and characterise Silica-Potassium-Phosphate (Si-K-P) solid fertiliser using sugarcane bagasse ash as a silica source. The process involved silica extraction using Potassium Hydroxide (KOH) followed by polymerization with Phosphoric Acid (H_3PO_4) under varying conditions of pH (5, 6, 7, 8, 9) and alkali silica concentrations (16.67%, 20%, 25%, 33.3%, and 50%). Characterisation methods included X-ray fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM). The results revealed that the raw sugarcane bagasse ash contained 69.2% silica (SiO_2). The synthesis conditions significantly influenced the final nutrient composition; the highest silica content of 22.8% was achieved at pH 7, the maximum potassium content of 59.9% at pH 9, and the highest phosphate content of 37.7% at pH 5. FTIR analysis confirmed the formation of siloxane ($Si-O-Si$), while SEM imaging showed a porous agglomerated morphology. This study demonstrates that sugarcane bagasse ash can be effectively converted into a value-added multinutrient fertilizer.

Contribution to Sustainable Development Goals (SDGs):
SDG 2: Zero Hunger
SDG 12: Responsible Consumption and Production
SDG 9: Industry, Innovation and Infrastructure
SDG 13: Climate Action

1. INTRODUCTION

1.1. Research Background

Indonesia is one of the world's largest sugarcane-producing countries, with production increasing year after year, which in turn drives sugar production. The sugar industry generates significant amounts of waste, including solid waste known as bagasse. In a single sugar production cycle, approximately 32% of bagasse is produced [1]. Bagasse from the milling process is often utilized as boiler fuel in the production process, substituting diesel and LPG. This utilization produces combustion waste known as sugarcane bagasse ash (SCBA) [2]. Generally, this ash is not used optimally and can have adverse environmental impacts; for instance, rainwater can carry it, disrupting drainage and causing floods, while fine dust particles can pollute the air and endanger human health. However, sugarcane bagasse ash has a sufficiently high silica (SiO_2) content of 69.2%, presenting an opportunity to utilize this waste as a raw material for silica-based composite fertilizer production.

Silica-based composite fertiliser is a fertiliser that combines silica with other essential plant nutrients. During the growth process, plants uptake silica (SiO_2) in addition to essential nutrients such as nitrogen (N), phosphorus (P), and potassium (K). Silica can enhance plant growth, photosynthesis, soil quality, and resistance to biotic and abiotic stresses. Silica-based composite fertilisers are particularly suitable for silica-accumulator plants that require substantial amounts of silica for growth, such as those inhe Gramineae family, including rice, sugarcane, bamboo, wheat, corn, and sorghum [3].

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Silica composite fertiliser is obtained by preparing a potassium silicate solution, which is then polymerised by adding an acid solution. Previous research has examined the morphology of $Si-K-N$ gel composites by extracting silica from rice husk ash using KOH and adding urea [4]. Given the potential of sugarcane bagasse ash, which has not been widely utilised despite its high silica content, this research aims to innovate by synthesising a composite fertiliser. Specifically, this study focuses on the "Synthesis and Characterization of Silica-Potassium-Phosphate ($Si-K-P$) Solid Fertilizer Derived from Sugarcane Bagasse Ash" to create a value-added product that provides essential nutrients like potassium and phosphate while utilizing industrial waste.

1.2. Literature Review

Synthesis of Silica Composite Fertilisers is generally ahieved through a chemical process involving the preparation of a potassium silicate solution, which is subsequently polymerised by the addition of an acid solution. This polymerization process is critical in forming the composite matrix that encapsulates nutrients. Various researchers have explored this method using different silica sources to optimize fertilizer morphology and nutrient content.

Previous Studies on Silica Sources and Formulations Study [4] examined the morphology of Silica-Potassium-Nitrogen ($Si-K-N$) gel composites utilizing rice husk ash as the silica source, extracted with a 2N Potassium Hydroxide (KOH) solution. By varying potassium silicate concentration, the research determined that the optimal composition was achieved at 1.2167% with the addition of 20 grams of urea. This formulation yielded a composite with a Silica content of 16.56% and a Potassium content of 82.49%.

In a different approach, [5] focused on synthesising a liquid potassium silicate fertiliser. The extraction process involved 30 grams of ash with various KOH concentrations and stirring speeds. The study concluded that the best liquid fertilizer was produced using 2.5N KOH at a stirring speed of 200 rpm, resulting in a product containing 10.93% Potassium and 1.5% Silica.

Furthermore, research [6] expanded into multi nutrient fertilizers using geothermal sludge waste. The study extracted silica using 2N KOH and performed acidification using a combination of nitric acid (HNO_3) and phosphoric acid (H_3PO_4). The findings highlighted the importance of pH in the synthesis process, stating that the optimal $Si-K-P$ solid fertilizer was formed at pH 5. This condition produced a fertilizer containing 27.5% Potassium and 13.61% Silica.

Research Gap and Novelty: Despite extensive research on rice husk, bamboo, and geothermal waste, the utilisation of Sugarcane Bagasse Ash (SCBA), which has a high silica content of approximately 69.2%, remains underutilised for solid composite fertiliser production. While previous studies [4][5][6] have successfully sysed $Si{-}K{-}N$ and liquid fertilisers, there is a specific need to develop a solid Silica-Potassium-Phosphate ($Si{-}K{-}P$) fertiliser derived from bagasse ash. Therefore, this research aims to synthesize and characterize this novel formulation, offering a value-added solution for sugar industry waste while providing a comprehensive nutrient source for agriculture.

Fig. 1. Pretreatment of SCBA : (A) Raw SCBA from factory, (B) SCBA after calcination

1.2.1. Extraction process

The extraction process serves as a critical foundational step in this research, determining the purity and quality of the precursor material used for fertilizer synthesis. Extraction involves separating a specific substance from a mixture by dissolving it in a suitable solvent. In the context of synthesizing silica-based fertilizers, the efficiency of silica recovery from waste materials is paramount. Previous studies have highlighted that successful silica extraction depends heavily on factors such as solvent type, concentration, temperature, and reaction time [7].

For silica extraction, the most effective method is alkaline dissolution. In this research, the silica (SiO_2) contained within the sugarcane bagasse ash is isolated by reacting the ash with a Potassium Hydroxide (KOH) solution. This reaction converts the insoluble silica into a soluble potassium silicate solution (K_2SiO_3), effectively separating it from carbon residues and other metallic impurities found in the ash. The chemical reaction governing this process is as follows [8] :

$$SiO_2 \quad + \quad 2KOH \quad \rightarrow \quad K_2SiO_3 \quad + \quad H_2O$$

This method is preferred over acid leaching because silica exhibits higher solubility in alkaline environments, resulting in a higher yield. Once the potassium silicate solution is obtained, it serves as the primary matrix for the subsequent polymerisation step, during which phosphate is introduced. The purity of the extracted silica directly influences the structural integrity and nutrient-release capabilities of the final $Si-K-P$ fertilizer [9].

1.2.2. Polymerization Process

The fundamental mechanism underlying the synthesis of the solid Si-K-P fertiliser is sol-gel polymerisation. This process involves the transition of a chemical system from a liquid "sol" (a colloidal suspension of solid particles) into a solid "gel" (a continuous inorganic network). In this study, the polymerization is initiated through the reaction between the silica precursor specifically potassium silicate derived from sugarcane bagasse ash and phosphoric acid (H_3PO_4) as the gelling agent.

The mechanism proceeds in two primary stages: hydrolysis and condensation. Upon the addition of phosphoric acid to the potassium silicate solution, an acid-base neutralization reaction

occurs. This reaction lowers the pH of the alkaline silicate solution, triggering the hydrolysis of silicate species to form silicic acid monomers (H_4SiO_4) and potassium phosphate salts. The stoichiometric reaction for this precursor formation can be described as follows [6] :

$$3(K_2SiO_3) \quad + \quad 3H_2O \quad + \quad 2H_3PO_4 \rightarrow \quad 2K_3PO_4 \quad + \quad 3H_4SiO_4$$

Following the formation of silicic acid monomers, the process advances to the condensation polymerization stage. In this phase, the unstable silanol groups ($Si-OH$) interact with one another to form stable siloxane bridges ($Si-O-Si$) while releasing water molecules as a byproduct. This condensation leads to the growth of silica chains, which eventually cross-link to form a rigid, three-dimensional porous network. The polymerization reaction is represented by the following equation [10] :

$$2Si(OH)_4 \rightarrow H_2(Si-O-Si)_2(OH)_4 + H_2O$$

Crucially, this polymerization does not result in pure silica. As the gel network forms, the potassium phosphate (K_3PO_4) species generated in the first step, along with excess potassium and phosphate ions, become physically entrapped or chemically adsorbed within the pores of the silica matrix. This encapsulation ensures that the final product is a composite material containing Silicon, Potassium, and Phosphate, rather than a simple physical mixture.

1.3. Research Objective

The objective of this study is to synthesize a solid Silica-Potassium-Phosphate ($Si-K-P$) composite fertilizer utilizing sugarcane bagasse ash as a silica precursor. This research specifically investigates the effect of key synthesis parameters—namely pH levels and alkali silicate concentrations—on the nutrient composition (SiO_2, K_2O, and P_2O_5) of the final product. Additionally, comprehensive characterisation using XRF, FTIR, and SEM is conducted to identify the optimal synthesis conditions for producing a high-quality fertiliser suitable for agricultural applications.

2. MATERIALS AND METHODS

2.1. *Materials and Equipment*

The primary raw material used in this experimental study was sugarcane bagasse ash (SCBA), a byproduct obtained from the boiler combustion unit of PT. PG Sinergi Gula Nusantara. For the chemical synthesis and extraction processes, specific reagents were employed. These included Potassium Hydroxide (KOH) in pellet form, which served as the alkaline solvent for silica extraction. Additionally, Phosphoric Acid (H_3PO_4) with a specific concentration of 85% was used as the phosphate nutrient source and as the neutralising agent to facilitate the polymerisation reaction. Distilled water (Aquadest) was utilized as the universal solvent for dissolving reagents. All chemical materials were purchased from UD. Nirwana and were used as received.

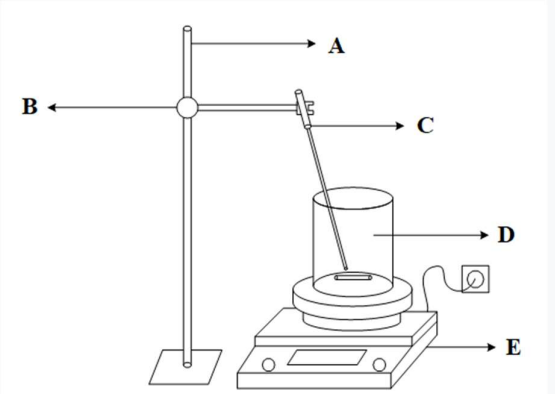

Fig. 2. Equipment for Extraction and polymerization

Description :
A. Retort stand
B. Clamp
C. Thermometer
D. Erlenmeyer flask
E. Magnetic stirrer

2.2. Methods

The research procedure consisted of four main stages : preparation of raw materials, silica extraction, polymerization of the Si-K-P fertilizer, and Aging and Characterization.

1. Preaparation of Raw Materials

First, the sugarcane bagasse ash collected at the factory underwent thermal pretreatment to improve its quality. The ash was calcined in a furnace at 800°C for 4 hours. This specific temperature and duration were chosen to fully decompose organic impurities, such as lignin and cellulose, ensuring that the remaining ash consisted primarily of high-purity inorganic silica [11]. Once the calcination was complete, the ash was cooled to room temperature before being processed in the extraction stage.

2. Silica Extraction

Silica was isolated from the prepared ash using an alkaline solid-liquid extraction method. A mass of 20 grams of the calcined ash was mixed with 200 ml of 2N Potassium Hydroxide (KOH) solution. The extraction reaction was carried out in a controlled environment at 100°C for 2 hours, with a constant stirring speed of 500 rpm. These operating conditions were optimized to maximize the dissolution rate of amorphous silica from the solid ash into the liquid phase, converting it into a soluble potassium silicate (K_2SiO_3) solution [8] [9]]. Upon completion of the reaction, the mixture was filtered through a filter paper to separate the potassium silicate filtrate from the insoluble solid residue (cake).

3. Polymerization of The Si-K-P Fertilizer

The resulting potassium silicate filtrate served as the main matrix for the fertilizer. This solution was diluted with distilled water to create various concentration ratios (50%, 33.3%, 25%, 20%, and 16.67%) to investigate the influence of silica content. The polymerization process was then initiated by slowly adding

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85% Phosphoric Acid (H_3PO_4) into the stirring silicate solution. The acid was added dropwise until specific pH targets were reached, ranging from pH 5 to 9. This step is critical as it neutralizes the alkali and triggers the condensation of silanol groups, forming a gel network that entraps the Potassium (K^+) and Phosphate (PO_4^{3-}) ions [6].

4. Aging and Characterization

To ensure the gel network was fully formed and stable, the synthesised mixture was aged for 24 hours at room temperature. Following this, the solid product was dried in an oven at 100°C for another 24 hours to remove moisture. The dried fertiliser was then crushed and sieved to achieve a uniform particle size of 100-mesh. Finally, the physicochemical properties of the fertiliser were analysed using X-Ray Fluorescence (XRF) for elemental composition, Scanning Electron Microscopy (SEM) for surface morphology, and Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups.

3. RESULT AND DISCUSSION

3.1. Analysis of Sugarcane Bagasse Ash

The raw material utilized in this study, specifically sugarcane bagasse ash calcined at 800°C, was subjected to X-Ray Fluorescence (XRF) analysis to ascertain its elemental and oxide composition. The quantitative data derived from this characterization are tabulated below :

Table 1. Analysis of Sugarcane Bagasse Ash

Chemical compounds	***Percentage (%)***
SiO_2	69.2
P_2O_5	2.6
SO_3	0.4
CaO	10.7
MnO	0.033
K_2O	9.02
Fe_2O_3	5.18
CuO	0.075

(Advanced Minerals and Materials Lab, Faculty of Mathematics and Natural Sciences, University of Muhammadiyah Malang, 2024)

3.2. Analysis of Si-P-K Content In the Product

Table 2. Analysis of Si-P-K Content In the Product

pH	potassium-silica concentration (%)	Composition (%)		
		Si	K	P
5	50%	14%	45.8%	37.7%
	33.30%	19%	47.30%	31.60%
	25%	22.7%	48.7%	25.5%
	20%	22.60%	49.80%	24.70%
	16.67%	22.5%	50.9%	23.9%
6	50%	18.40%	51.15%	31.35%
	33.30%	20.20%	49.40%	27.68%
	25%	21.85	47.45%	24%
	20%	22%	53%	23.68%
	16.67%	22.30%	51.60%	23.35%
7	50%	22.8%	49.1%	25%
	33.30%	21.90%	51.50%	23.75%
	25%	21%	53.60%	22.5%
	20%	21.40%	58%	22.65%
	16.67%	22.1%	52.3%	22.8%
8	50%	21.35%	56.75%	23.45%
	33.30%	19.45%	54.50%	23%
	25%	17.50%	52.10%	22.55%
	20%	18.10%	51.40%	22.73%
	16.67%	19.05%	54.15%	22.90%
9	50%	19.9%	59.9%	21.9%
	33.30%	17%	57.50%	22.25%
	25%	14%	55.1%	22.6%
	20%	14.80%	55.50%	22.80%
	16.67%	16%	56%	23%

(Source: Integrated Research and Testing Laboratory, UGM)

The chemical composition of the synthesised Si-K-P solid fertiliser was determined by X-ray fluorescence (XRF) analysis to verify the successful incorporation of key nutrients. The quantitative results, as presented in Table 2, reveal the oxide percentages of Silicon (SiO_2), Potassium (K_2O), and Phosphate (P_2O_5) across various synthesis conditions. These data provide a comprehensive overview of how the interplay between pH levels and alkali silicate concentrations directly impacts the final nutrient formulation of the composite fertilizer.

3.1. *Effect of pH Polimeryzation and potassium silicate Concentration on SiO_2 levels*

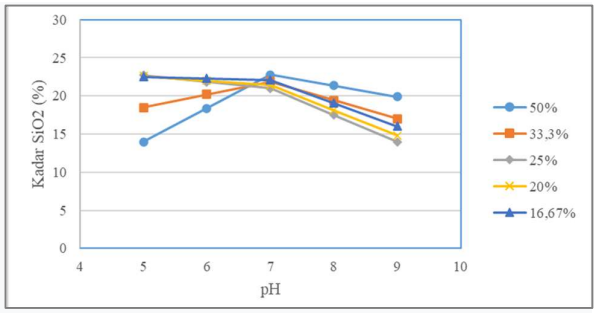

Fig. 3. Relation Between Concentrations of potassium silicate solution (%) and SiO_2 levels, at Various pH

Based on the quantitative data presented in Table 2, the silica content exhibits a non-linear response to pH adjustments, indicating a specific stability peak. The analysis reveals that the optimum silica content of 22.8% was achieved under neutral conditions (pH 7) with a 50% alkali silicate concentration. Deviating from this neutral point—whether towards acidic (pH 5) or alkaline (pH 9) conditions—resulted in a noticeable decline in silica retention within the solid matrix. This empirical evidence suggests that a neutral environment serves as the critical threshold for maximising the yield of the silica network in the final fertiliser product.

Theoretically, this phenomenon is governed by the kinetics of the sol-gel polymerization process. As elucidated by [6], the rate of silanol condensation reaches its maximum efficiency at a neutral pH. In this state, the equilibrium between the hydrolysis of the precursor and the condensation of silanol groups ($Si-OH$) is optimized, facilitating the rapid formation of stable siloxane bridges ($Si-O-Si$). At this thermodynamic "sweet spot," the solubility of silica species is minimized, allowing the polymer network to precipitate effectively. Conversely, under highly alkaline or acidic conditions, the stability of soluble silicate species increases, thereby inhibiting the complete gelation and precipitation of the solid silica matrix.

3.2. *Effect of pH Polimeryzation and potassium silicate Concentration on SiO_2 levels*

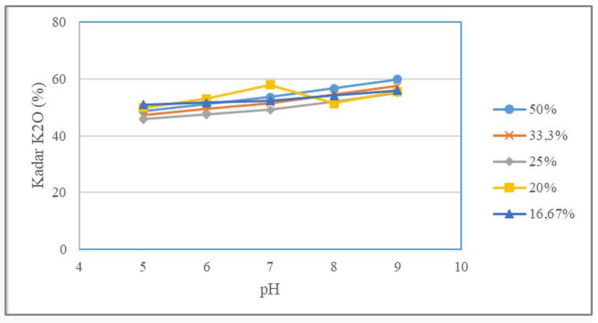

Fig. 4. Relation Between Concentrations of potassium silicate solution (%) and K_2O levels, at Various pH

Regarding potassium loading, the experimental results show a clear positive linear correlation between the pH of the synthesis medium and the final potassium concentration. As detailed in the table, the percentage of Potassium Oxide (K_2O) rises progressively with increasing alkalinity, reaching a substantial peak of 59.9% at pH 9 (50% concentration). This trend indicates that an alkaline environment significantly enhances the fertiliser matrix's capacity to retain potassium ions, whereas acidic conditions (pH 5) result in considerably lower potassium incorporation.

This behaviour can be scientifically attributed to changes in the surface charge of the silica matrix. According to [12], the silica surface undergoes deprotonation as the pH increases above its Point of Zero Charge (PZC), leading to the formation of negatively charged silanolate sites ($Si-O^-$). These anionic sites generate a strong electrostatic attraction towards the positively charged Potassium cations (K^+) present in the solution. Consequently, the high pH conditions act as a catalyst for ion adsorption, promoting the extensive entrapment of potassium within the developing silica structure.

3.3. *Effect of pH Polimeryzation and potassium silicate Concentration on SiO_2 levels*

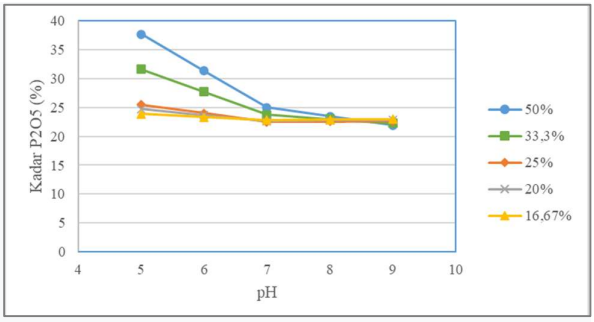

Fig. 5. Relation Between Concentrations of potassium silicate solution (%) and K_{2O} levels, at Various pH

In direct contrast to the potassium trend, the phosphate content shows a strong inverse relationship with pH. The data highlights that the maximum Phosphorus Pentoxide (P_2O_5) concentration of 37.7% was recorded at the most acidic condition (pH 5) with a 50% concentration. As the synthesis environment becomes more alkaline (moving towards pH 9), a significant reduction in phosphate content is observed. This confirms that acidic conditions are the primary driver for maximizing phosphorus loading in the $Si-K-P$ composite.

The theoretical underpinning for this result involves both stoichiometric and electrostatic factors. Chemically, achieving a lower pH necessitates the addition of a larger volume of Phosphoric Acid (H_3PO_4) to neutralize the alkaline precursor, thereby directly increasing the molar ratio of phosphate available for reaction. Furthermore, [13] explains that at higher pH levels, the silica surface becomes predominantly negative, creating an electrostatic repulsion against the anionic phosphate species (PO_4^{3-}). By maintaining an acidic environment (pH 5), this repulsion is reduced, enabling more efficient incorporation of phosphate ions into the porous silica network.

3.4. *Analysis of SEM Results*

Following chemical and functional group characterisation, the structural topography of the synthesised fertiliser was further investigated using Scanning Electron Microscopy (SEM). This imaging technique was used to visually evaluate the surface morphology, particle agglomeration, and porosity of the Si-K-P

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composite produced by the sol-gel process. The SEM micrographs revealing the surface structure are presented in the figure below:

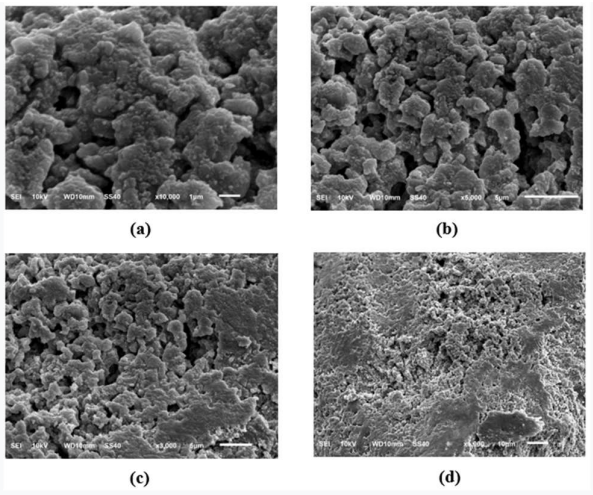

Fig. 6. SEM Analysis Results on Samples with pH 7 and Potassium Silica Concentration of 50% : (a) Magnification 10000× (b) Magnification 5000× (c) Magnification 3000× (d) Magnification 1000×

The analysis focused on the optimal sample synthesized at pH 7 with a 50% alkali silicate concentration. The SEM micrographs, captured at magnifications ranging from 1000x to 10000x, reveal a highly heterogeneous and non-uniform surface structure. At lower magnifications, the fertilizer appears as large, irregular agglomerates with a rough surface texture, rather than distinct individual particles. These agglomerates are composed of primary particles that have fused together to form a solid, continuous network. Furthermore, the microstructure exhibits significant porosity, characterised by voids within the agglomerated masses, a typical feature of materials synthesised via the sol-gel precipitation method.

The kinetics of the polymerisation process fundamentally governs the formation of this highly agglomerated, porous structure. As described by [6], a neutral environment (pH 7) represents the point of minimum stability for the silicate solution, where the rate of silanol condensation (gelation) is at its maximum. This rapid gelation, combined with the high solute availability from the 50% concentration precursor, results in instantaneous, uncontrolled precipitation. Consequently, the primary silica particles fuse immediately upon formation before they can grow into well-defined, separate grains. The observed irregular, agglomerated morphology also strongly indicates the material's amorphous nature, consistent with [14], which found that silica derivatives produced via sol-gel routes predominantly exhibit non-crystalline structures. Additionally, the fused particles observed at high magnification are within the micrometre range, consistent with the definition of microparticles[15].

3.5. Analysis of FTIR Results

To definitively identify the chemical bonds and functional groups present in the fertiliser matrix, Fourier Transform Infrared Spectroscopy (FTIR) was performed. This characterisation serves to chemically validate the success of the polymerisation reaction involving the silica precursor, potassium hydroxide, and phosphoric acid. The FTIR spectrum of the optimum sample (synthesized at pH 7 with 50% concentration) is presented in the Figure 7 below :

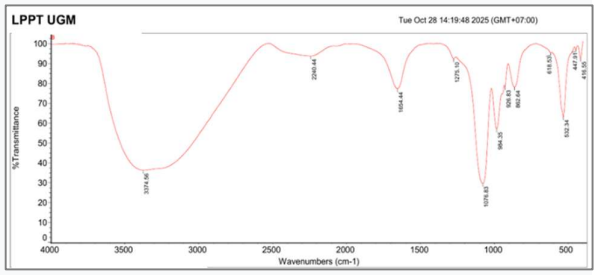

Fig. 7. FTIR Analysis Results on Samples with pH 7 and Potassium Silica Concentration of 50%

As shown in Figure 5, the functional group characterization of the fertilizer synthesized at the optimal condition (pH 7 with 50% alkali silicate concentration) was performed using FTIR spectroscopy. This analysis is crucial to validate the chemical transformation of the raw materials into a solid $Si-K-P$ composite.The spectrum begins with a broad absorption band in the high-energy region, centred at 3374.56 cm^{-1} (spanning 3600–3000 cm^{-1}). This broadening is a classic indicator of hydroxyl group ($-OH$) stretching vibrations. In the context of this research, this peak originates from two simultaneous sources the presence of silanol groups ($Si-OH$) that are integral to the silica surface, and water molecules ($H-O-H$) that are physically trapped or adsorbed within the fertilizer's pores. The presence of moisture is further corroborated by a smaller, sharper peak at 1654.44 cm^{-1}, which corresponds to the bending vibration of water molecules ($H-O-H$). These findings, which align with [16] and [17], suggest that the resulting ferser exhibits hygroscopic properties that help maintain soil moisture.

Moving to the "fingerprint" region of the spectrum, the most dominant and defining feature is the intense peak observed at 1076.83 cm^{-1}. According to [17], this strong absorption is the signature of asymmetric stretching vibrations of siloxane bonds (Si-O-Si). The emergence of this peak is the primary evidence that the silicic acid monomers have successfully undergone polymerization to form a robust, cross-linked inorganic framework. Additionally, the spectrum displays peaks at 984.35 cm^{-1} and a shoulder at 926.83 cm^{-1}, which are attributed to the stretching vibrations of remaining silanol groups ($Si-OH$). These groups represent the active sites on the silica gel surface that facilitate further chemical interactions.

Finally, the analysis confirms the integration of nutrients, specifically Potassium and Phosphate. Although potassium ions (K^+) are monoatomic and do not produce direct infrared signals, their existence is chemically linked to phosphate compounds. Given the high nutrient content detected via XRF (49.1% K and 25% P), the intense peak at 1076.83 cm^{-1} is understood to be a superposition—an overlap between the silica vibrations and the phosphate bond ($P-O$) vibrations found in potassium phosphate salts [17]. This interpretation is strongly supported by the presence of minor peaks at 618.53 cm^{-1} and 532.34 cm^{-1}. These specific bands are characteristic of phosphate salts, effectively confirming that both Potassium and Phosphate have been chemically incorporated into the silica matrix [6].

4. CONCLUSION

The findings of this study confirm the viability of sugarcane bagasse ash as a silica precursor, yielding a silica content of 69.2% following thermal treatment. By employing alkaline extraction and subsequent acid polymerization, a solid Silica-Potassium-Phosphate ($Si - K - P$) fertilizer was successfully synthesized. The experimental results indicate that synthesis conditions significantly govern the nutrient composition of the final product. Specifically, a neutral pH (7) was optimal for silica retention (22.8%), while alkaline conditions (9) maximised potassium content (59.9%), and acidic conditions (5) favoured phosphate incorporation (37.7%). Furthermore, characterisation by FTIR and SEM confirmed the formation of a porous siloxane network encapsulating the nutrients, thereby verifying the successful synthesis of the composite fertiliser.

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