

Eco-Friendly PVA/Starch/Rice Husk Char Coating For Controlled Release Fertilizers

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ABSTRACT

This research explores the development of a novel slow-release fertilizer coating composed of polyvinyl alcohol (PVA), tapioca starch, and modified rice husk char. The study focuses on the optimal production of rice husk char at 600°C, which facilitates enhanced silica and reduced carbon contents, improving its functional properties in the coating matrix. The incorporation of rice husk char into the PVA/starch blend significantly alters the film's structural and chemical characteristics, as confirmed by FTIR analysis, which showed increased Si-O bonding. The coatings effectively reduced moisture absorption by approximately 50% in comparison to uncoated di-ammonium phosphate (DAP) granules, demonstrating superior protective qualities. Additionally, nutrient release profiles indicated a controlled release over 24h, which is critical for reducing environmental leaching. These results underscore the potential of using rice husk char in biopolymer coatings to enhance the environmental performance of fertilizers, offering a sustainable approach to agricultural management.

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1. Introduction

The use of fertilizers plays a crucial role in improving crop yields and meeting the growing food demand of an ever-increasing global population. However, the excessive and inefficient application of conventional fertilizers can lead to substantial nutrient losses, environmental pollution, and detrimental effects on human health [1], [2]. To mitigate these issues, controlled-release or slow-release fertilizers (SRFs) have emerged as a promising solution, offering a more efficient and environmentally friendly approach to nutrient management [3].

Slow-release fertilizers are designed to release nutrients gradually over an extended period, reducing the risk of nutrient leaching, volatilization, and environmental contamination [4]. These fertilizers can be broadly categorized into two main types: non-coated SRFs, which rely on inherent chemical or physical properties to control the release rate, and coated SRFs, which employ protective coatings or encapsulation techniques to regulate nutrient release [5].

Among the various coating materials explored for SRF development, polymers have garnered significant attention due to their ability to form semi-permeable membranes that control nutrient diffusion [6]. However, conventional petroleum-based polymers raise concerns due to their non-biodegradability and potential environmental impact. As a result, there is a growing interest in developing biodegradable and eco-friendly polymer coatings derived from renewable and sustainable sources [7].

Biopolymers such as starch, polyvinyl alcohol (PVA), and natural fibers have emerged as promising candidates for SRF coatings owing to their biodegradability, low toxicity, and environmental compatibility [8], [9]. Starch, a naturally abundant and inexpensive biopolymer, has been extensively studied for its potential in controlled-release applications due to its film-forming ability and biodegradability [10]. When combined with other biopolymers like PVA, starch can form hybrid coatings with improved mechanical strength and controlled permeability [11]. Additionally, the

incorporation of natural fillers like rice husk ash can further enhance the coating properties and potentially provide additional benefits, such as adsorption or ion-exchange capabilities [12].

Rice husk, a byproduct of rice milling, is an abundant and underutilized agricultural residue. It is composed primarily of organic compounds like cellulose, hemicellulose, and lignin, along with a significant amount of inorganic silica (around 20%) [13]. By subjecting rice husk to controlled pyrolysis or combustion, the organic components can be removed, leaving behind a carbon-rich material known as rice husk char or ash, which retains a high silica content [14]. The silica present in rice husk char possesses unique properties, including a high surface area, porous structure, and potential for ion-exchange and adsorption. These characteristics make rice husk char an attractive filler material for SRF coatings, as it can impart controlled porosity, enhance nutrient adsorption, and potentially improve the overall performance and longevity of the coating system [12].

This research aims to develop an eco-friendly and biodegradable coating system for slow-release fertilizers by combining starch, PVA, and rice husk char. The proposed coating formulation leverages the unique properties of each component to create a multi-functional barrier that regulates nutrient release while offering environmental compatibility and potential value-added benefits.

2. Materials and Methods

Preparation of Biochar from Rice Husk

Rice husk was first thoroughly washed with water and naturally air-dried, followed by oven-drying at 100°C for 2 h. The dried husks were then placed into sealed 304 stainless steel tubes (42 mm diameter, 150 mm length) with clay-sealed caps to limit oxygen exposure. The tubes were heated in a Nabertherm furnace (Germany) to examine the effects of temperature on the biochar quality. The temperature settings explored were 500°C, 600°C, 700°C, and 800°C with a heating rate of 400°C/h and a dwell time of 4 h at the peak temperature. After treatment, the obtained biochar was cooled and ground.

Extraction of Silica from Rice Husk Biochar

Silica was extracted from the biochar using alkaline treatment to convert it into soluble sodium silicate, facilitating plant absorption. Three grams of biochar were added to 30 mL of sodium hydroxide (NaOH) solution at concentrations of 0.1 M, 0.5 M, 1.5 M, 2 M, and 2.5 M. The mixture was shaken intermittently at 150 rpm for 6 h (three sessions of 2 h each, with 2-hour intervals) and then left to stand for 24 h. Post-extraction, the mixture was centrifuged at 7168 rcf for 5 minutes and filtered to separate the undissolved biochar. The filtrate was diluted and the effective silicon content was measured using UV-Vis molecular absorption spectroscopy according to TCVN 11407-2019 at 800 nm wavelength. The biochar residue was further analyzed using Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM).

Acid Hydrolysis of Fused Phosphate Fertilizers

Fused phosphate fertilizers were reacted with sulfuric acid (H₂SO₄), hydrochloric acid (HCl), and nitric acid (HNO₃) in varying equivalent concentrations. Ten grams of fused phosphate was treated with 100 mL of each acid solution at 0.5 N, 1.0 N, and 2.0 N dilutions. The samples were agitated intermittently at 150 rpm for 6 h (three sessions of 2 h each, with 2-hour intervals) and then left to rest for 24 h. Following incubation, the mixtures were centrifuged at 7168 rcf for 5 minutes and filtered. The P₂O₅ content in the filtrates was determined using UV-Vis spectroscopy according to TCVN 10678:2015 at a wavelength of 831 nm.

Preparation of PVA/Starch/Biochar Slow-Release Fertilizer Coating

Starch (7 g) was mixed with 70 mL of distilled water and heated to 90°C at 500 rpm for 1 hour to achieve complete gelatinization. Separately, PVA (3 g) was swollen under stirring in 30 mL of water for 10 minutes, then heated to 80°C for 2 h until completely dissolved. Both mixtures were cooled to room temperature (~30°C), combined, and stirred at 60°C for 2 h to form a homogeneous PVA/starch mixture.

Rice husk char (3 g) was mixed with 100 mL of 1 M NaOH and shaken horizontally at 150 rpm for 6 h (three 2-hour intervals with 2-hour breaks in between), followed by resting for 24 h. The mixture was then neutralized and stirred for 1 hour to form a gel with a neutral pH (6-8). This gel was added to

four separate PVA/starch blends above so that the mass of char was 3%, 6%, 9%, and 12% of the (PVA+starch) solid mass (correspondingly labeled as C3, C6, C9, and C12, respectively. A control sample (C0) contained no biochar. To initiate crosslinking, 5 mL of 0.25 M ammonium persulfate (APS) solution was added to each blend, and the mixtures were stirred at 500 rpm at 60°C for 1 hour, then heated to 80°C to promote the reaction between the starch and PVA over 2 h. Subsequently, 0.5 mL of formaldehyde solution was added, and the mixture was stirred at 500 rpm at 80°C for 3 h to form crosslinks in the film. Finally, 1 mL of glycerol was mixed in and stirred for 2 h [15].

The final mixtures were sonicated to remove air bubbles and cooled to about 30°C. A portion of each mixture was poured into plastic Petri dishes lined with silicone paper to dry naturally, forming the films. The remainder was stored in airtight containers for use as a coating for DAP fertilizer granules.

Coating DAP fertilizer granules

A drying oven and plastic Petri dishes lined with silicone paper were prepared. Two grams of size-sorted and oven-dried DAP granules were placed into a 50 mL glass beaker. The granules were quickly dipped into the coating mixture for 1 second, touched against the beaker walls three times to remove excess coating, and then partially dried using a handheld hair dryer. After coating all the granules, they were placed in an oven at 60°C for 1 hour. This process was repeated twice more. After the third application, the coated granules were dried for 2 h at 60°C.

Moisture Absorption Study

The moisture absorption capacity (MAC) of both coated and uncoated fertilizer was assessed. A specific quantity of fertilizer was weighed (m_0) and placed on a pre-weighed dry Petri dish. The setup was then placed in a sealed container with a saturated NaCl solution, maintaining a relative humidity (RH) of 75%. After 12 h, the fertilizer was weighed again (m_1). The moisture absorption level was calculated using the formula:

$$MAC(\%) = \frac{m_1 - m_0}{m_1} \times 100 \quad (1)$$

Nutrient Release Study in Water

The release of nutrients from the fertilizer, specifically phosphorus pentoxide (P_2O_5), was evaluated for both coated and uncoated samples. Two grams of each type of fertilizer were placed in 100 mL of distilled water. The mixture was agitated at 150 rpm on a horizontal shaker. Every 2 h, a 1 mL sample was withdrawn and analyzed for dissolved P_2O_5 concentration according to TCVN 10678:2015. This process continued for a duration of 24 h.

3. Results and Discussion

3.1. Silica extraction from rice husks

3.1.1. Effect of Temperature on Rice Husk Char Properties

The formation of rice husk char via pyrolysis in an anaerobic environment showed significant dependence on temperature, particularly affecting the carbon and silicon content of the char. Preliminary compositional analysis via Energy Dispersive X-ray Spectroscopy (EDS) indicated distinct variations in elemental composition with temperature changes (Table 1).

Table 1. Elemental composition of rice husk char at different temperatures.

Temperature (°C)	C	O	Si	K
Raw Husk	52.63 ± 1.99	43.33 ± 1.69	1.71 ± 1.94	0.47 ± 0.10
500	78.88 ± 21.52	28.18 ± 19.74	2.78 ± 2.70	0.70 ± 0.69
600	65.55 ± 9.87	26.67 ± 7.82	7.06 ± 2.89	0.44 ± 0.21
700	65.42 ± 18.97	27.51 ± 14.30	6.28 ± 4.57	0.42 ± 0.18
800	48.13 ± 3.34	40.47 ± 2.82	10.68 ± 1.02	0.63 ± 0.12

As the temperature increased to 500°C, the oxygen content decreased while the content of other elements increased possibly due to the loss of water. As the temperature continued to rise to 800 °C, the carbon content decreased because of the release of CO₂ and other organic compounds from the breakdown of the cellulosic components in the rice husk (cellulose, hemicellulose, and lignin) [16]. Importantly, the silicon content increased with higher temperatures, because silicon does not form volatile compounds that would be lost.

In the next stage of silica extraction, the silica content in the char should be high and the carbon content should be low. This allows NaOH to effectively access the silica. However, after the silica is extracted, the remaining solid will be mainly carbon, which contribute to the composition of the fertilizer coating. So a high carbon content in the char after pyrolysis is also desirable. To balance these competing factors, the researchers chose 600°C as the optimal pyrolysis temperature. This allowed for efficient silica extraction while also retaining a useful amount of carbon in the final char.

3.1.2. Silica Extraction from Rice Husk Char Using NaOH

The extraction of silica from the rice husk biochar was based on the following reaction:



Fig. 1 shows that the silica extraction efficiency increased with NaOH concentration, plateauing beyond 1.0 M, indicating minimal additional benefits from higher concentrations. The reason for this plateau is that when silica in the char dissolved enough to form a silicate solution with a moderate concentration, the high viscosity of the silicate-rich mixture made it increasingly difficult to dissolve any further silica. Thus, 1 M NaOH was determined to be the most effective concentration for silica extraction.

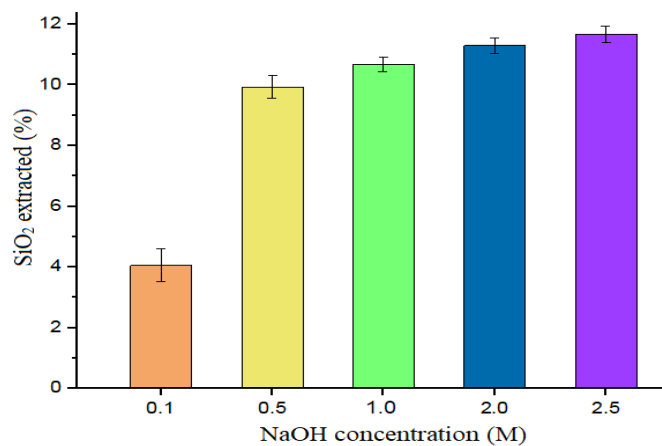


Figure 1. Effect of NaOH concentration on percentage of SiO₂ extracted from rice husk char.

3.1.3. SEM and FTIR Analysis of NaOH-Treated Rice Husk Char

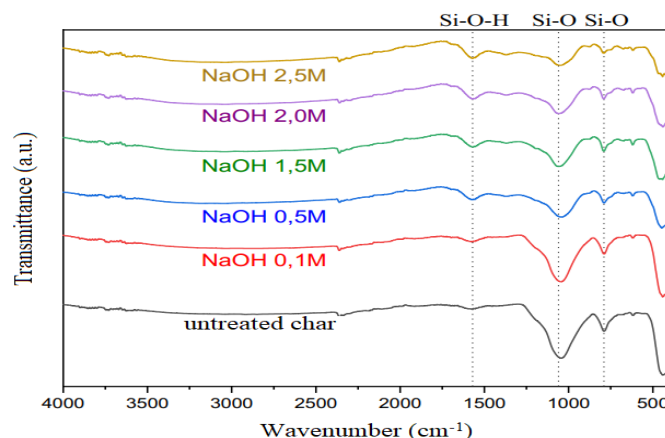


Figure 2. FTIR spectra of untreated biochar and biochar after treated with different NaOH concentrations.

During the extraction of silicate from rice husk ash, structural modifications and functional group changes were observed. To assess these changes, Fourier Transform Infrared Spectroscopy (FTIR) was employed across a wavelength range of 4000 cm^{-1} to 400 cm^{-1} to identify alterations in specific bonds (Fig. 2). The presence of a Si-O bond was indicated by peaks approximately at 793.33 cm^{-1} and 1055.3 cm^{-1} , while the peak at 1650 cm^{-1} corresponded to O-H bonds in adsorbed water molecules and silanol groups [17].

Fig. 2 displays that the increase in concentration of NaOH led to a gradual decrease in the peak intensities of Si-O bonds and an increase in those of Si-O-H bonds. This suggests that as NaOH concentration increases, the silica content in the rice husk ash powder decreases.

Scanning Electron Microscopy (SEM) was used to observe the structural changes in the surface of rice husk ash before and after modification with various concentrations of NaOH. Figure 3a depicts the original surface structure of the rice husk char, which is characterized by clear framework outlines and few pores. However, Fig. 3b shows partial destruction of this structure, with a loss of the surface contours. In Fig. 3c, these contours were almost lost, and numerous pores appear on the surface. The loss of contours and appearance of pores are attributed to the strong dissolution of silica at high NaOH concentrations [18].

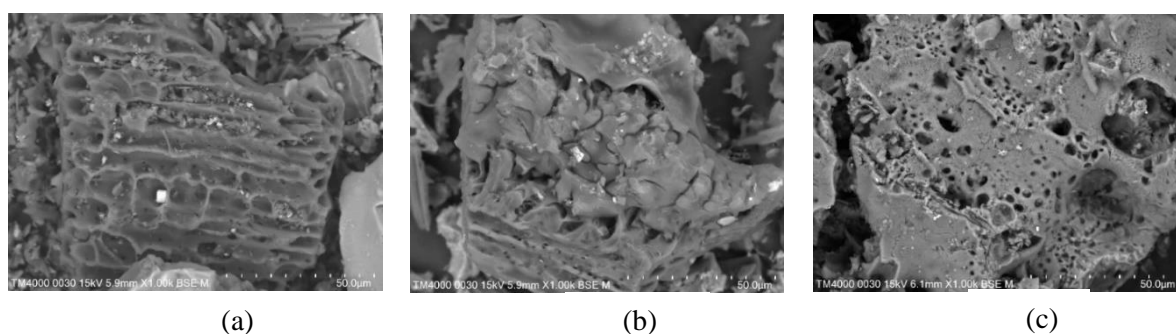


Figure 3. SEM micrographs of rice husk biochars (a) untreated, (b) treated with 0.1 M NaOH (c) and treated with 1.0 M NaOH.

3.2. Effect of Acid Concentration on Phosphate Extraction

Phosphate extraction from fused phosphate fertilizers was influenced by the type and concentration of acid used, with HCl and HNO₃ showing superior dissolution capabilities compared to H₂SO₄ (Table 2). The use of 2 N HNO₃ was favored due to its ability to dissolve the highest amount of P₂O₅ and provide nitrogen to the plants. A more concentrated HNO₃ may dissolve more P₂O₅, but the low volume required to neutralize the fused phosphate fertilizer would make the mixture too viscous to agitate effectively. These findings support the potential use of extracted phosphate solutions in combination with silica gel from rice husk char for developing slow-release fertilizer coatings.

Table 2. Percentage of P₂O₅ dissolved from phosphate fertilizer by acid type and concentration.

Concentration (N)	H ₂ SO ₄	HCl	HNO ₃
0.5	1.77 ± 0.85	2.55 ± 0.69	1.82 ± 0.72
1.0	1.94 ± 0.72	4.30 ± 0.71	3.69 ± 0.63
2.0	3.68 ± 0.89	4.99 ± 0.89	4.97 ± 0.82

After hydrolyzing fused phosphate fertilizer, we obtained a solution with a high P₂O₅ content. This solution was used to neutralize the mixture of rice husk char in NaOH. The resultant mixture comprised poorly soluble salts, specifically MeNH₄PO₄·H₂O (where Me represents Mg, Fe, Zn, Mg, or K) and silicon-phosphate gel (Si₃(PO₄)₄). Some forms of silica that exhibit strong binding capabilities with phosphate show potential for application as coatings in slow-release fertilizers [19].

3.3. Development of DAP Fertilizer Coatings

3.3.1. Appearance and moisture absorption by coated DAP granules

The dip coating method successfully produced a coating on DAP fertilizer granules. By using gravimetric method, we found that the coating layer comprised of 20% of the granule weight. The uniformity of the coating was confirmed by the appearance of the granules in sequential images (Fig. 4a to 4f), showing a gradual increase in char content from 0% to 12% in the coating layer.

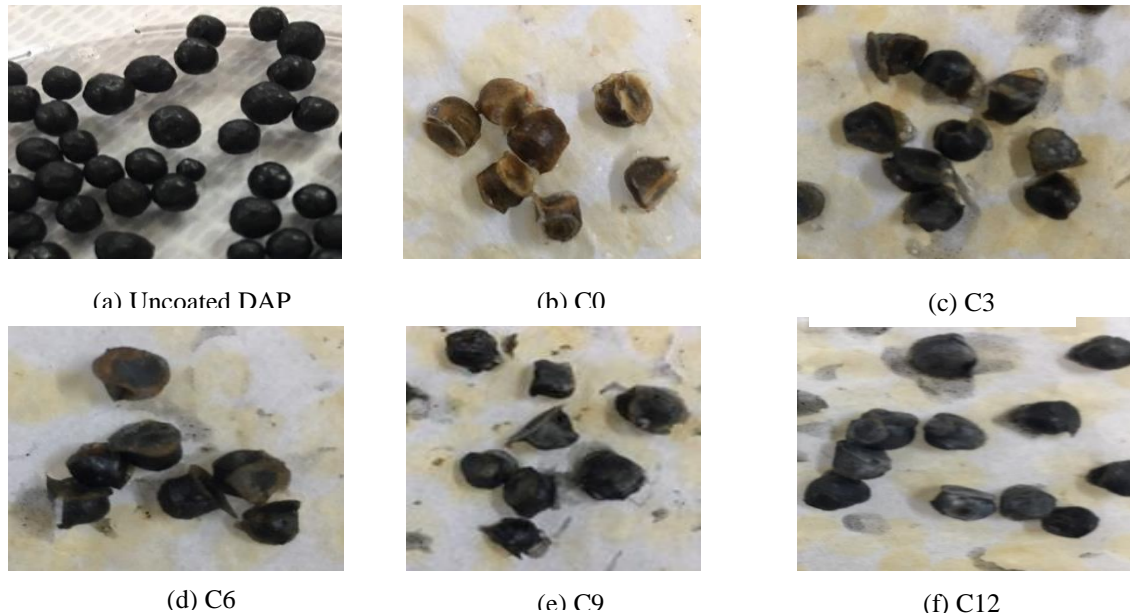


Figure 4. Uncoated (a) and coated (b-f) DAP granules with different content of rice husk char in the coating layer.

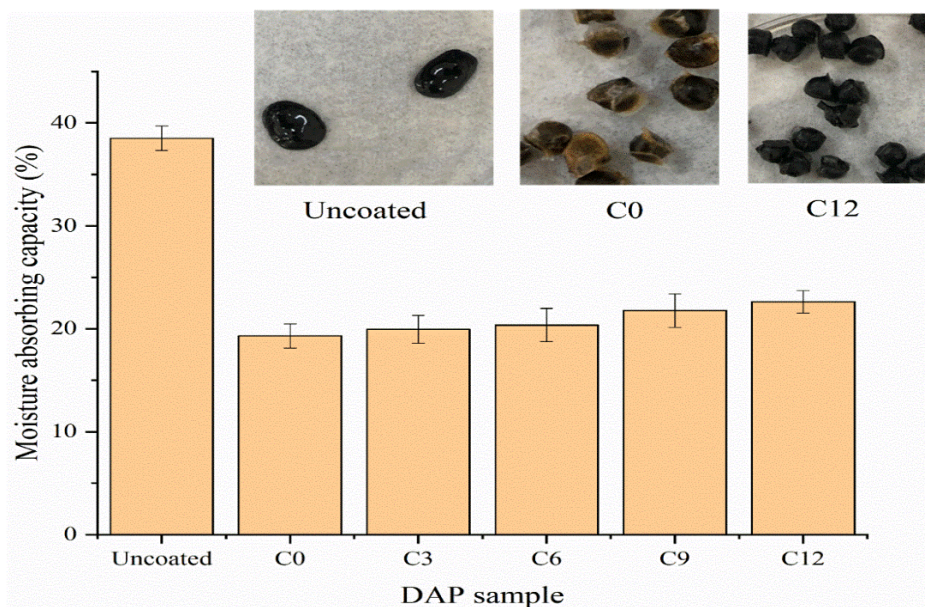


Figure 5. Moisture absorption of uncoated and coated DAP granules with different char content. Inset: appearance of uncoated, C0, and C12 granules.

The moisture absorption after 12 h was measured to assess the protective quality of the coatings. The inset in Fig. 5 shows that the uncoated DAP granules deliquesced while the coated granules still kept their form. The quantitative results in Fig. 5 demonstrated that uncoated granules absorbed significantly more moisture compared to coated ones, demonstrating the effectiveness of the PVA/starch/char coatings in reducing moisture uptake.

The nutrient release rate, specifically phosphorus (P_2O_5), from uncoated and coated DAP granules was monitored over 24 h. The results highlighted a slower release rate from coated granules compared to the uncoated ones (Fig. 6). Moreover, increasing the char content in the coating layer resulted in an increase in the release rate due to the high porosity of the char, which was revealed by the SEM results. This result implies we can control the release rate of nutrients from DAP by changing the char content in the coating.

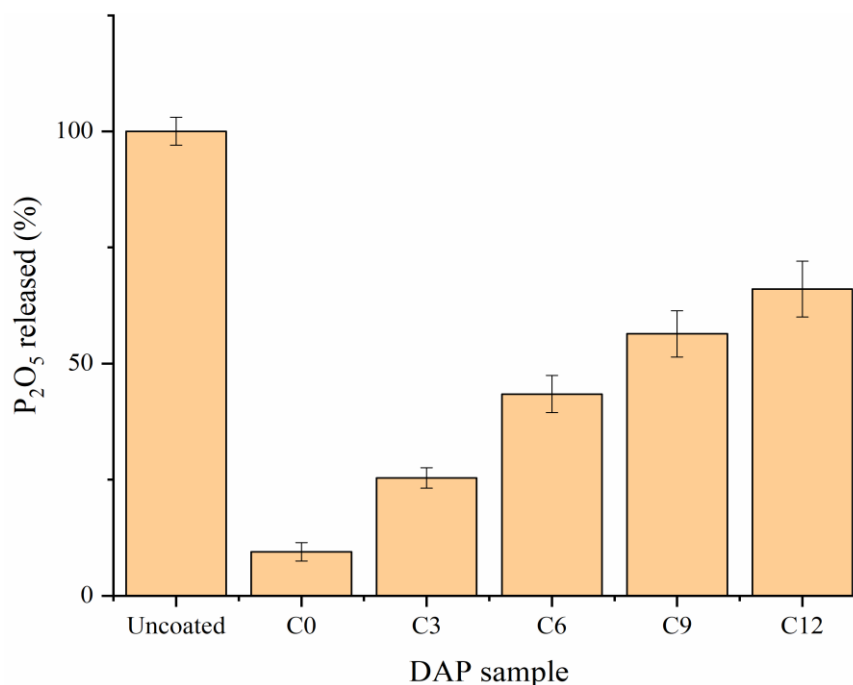


Figure 6. Percentage of P_2O_5 released from uncoated and coated DAP granules with different char content.

4. Conclusions

This study demonstrated the successful development and characterization of a sustainable, slow-release fertilizer coating made from PVA, tapioca starch, alkaline-treated rice husk char, and acid-treated fused phosphate fertilizer. The optimal pyrolysis temperature for rice husk char production was identified as $600^{\circ}C$, which maximized the silica content while minimizing carbon content, enhancing the decomposition process. The modified char, when incorporated into the PVA/starch matrix, significantly influenced the film's properties by increasing silicon linkage and adjusting the structural integrity, as evidenced by FTIR spectral shifts.

The coating formulations effectively reduced the moisture uptake by up to 50% compared to uncoated DAP granules, with increased char content improving the barrier properties. Nutrient release studies showed that the coated fertilizers had a controlled release pattern, which could potentially reduce nutrient loss and increase efficiency in agricultural applications. Overall, the findings suggest that integrating rice husk char into biopolymer-based coatings could offer an eco-friendly solution to improving fertilizer performance and environmental sustainability.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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