

Effects of Stirring Speed and Impregnation Duration on Phosphate Impregnation and Physicochemical Properties of Rice Husk Biochar

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Abstract

Efficient phosphate fertilization remains challenging due to the limited bioavailability of phosphorus in soil and losses from conventional phosphate fertilizers. This study investigated the effects of stirring speed (600 and 1200 rpm) and impregnation duration (6–30 h) on phosphate impregnation and mass transfer into rice husk-derived biochar using monoammonium phosphate (MAP). Proximate composition and acid- and water-soluble P content were analyzed to assess nutrient retention and diffusion behavior. Impregnation duration exerted the strongest influence, producing progressive reductions in moisture and volatile matter and increased ash content across all treatments. Water- and acid-soluble P increased consistently with longer contact time, with maximum values of $14.350 \pm 0.450\%$ and $15.787 \pm 0.186\%$ (mean $\pm$ SD), respectively, at 1200 rpm and 30 h. Mass transfer rates were highest during the initial 6 h, followed by a sharp decline as adsorption sites became saturated and diffusion resistance increased. While higher stirring speed moderately improved early mass transfer, its effect diminished at extended durations. These findings indicate that phosphate impregnation in rice husk biochar is primarily governed by impregnation duration, with stirring speed contributing only a modest enhancement. Further release kinetics studies are required to determine whether the resulting phosphate-impregnated biochar exhibits slow-release fertilizer behavior.

Keywords: biochar, impregnation, mass transfer, phosphate-impregnated, rice husk

1. Introduction

As an essential macronutrient, phosphorus (P) supports plant growth by participating in energy conversion, intracellular communication, and photosynthetic activity. In agricultural systems, phosphorus is generally supplied through phosphate fertilizers, which provide orthophosphate ions (H_2PO_4^- and HPO_4^{2-}) as the main source (El-Hamady, 2016; Yasmin *et al.*, 2004). However, much of the applied P becomes immobilized through adsorption and fixation by calcium, iron, and aluminium oxides. These reactions reduce fertilizer efficiency, hinder plant uptake, and lead to substantial nutrient losses (Prasad *et al.*, 2022; Zhong *et al.*, 2018). Excessive fertilizer application to compensate for these losses often leads to runoff-driven eutrophication, algal blooms, and oxygen depletion in aquatic ecosystems (Chen *et al.*, 2023; Logesh Kumar *et al.*, 2024). These issues highlight the urgent need for fertilizer technologies that enhance P use while minimizing environmental losses.

Biochar has emerged as a promising material for improving nutrient retention and supporting controlled-release fertilization because of its high porosity, diverse surface functional groups, and ion-exchange properties that facilitate nutrient adsorption and stabilization. Rice husk represents an abundant and underutilized biochar feedstock in Indonesia, which produced 54.75 million tons of rice in 2022, with East Java contributing 9.69 million tons (BPS-Jatim, 2022; BPS, 2022). Its lignocellulosic composition is suitable for biochar production and favorable for nutrient enrichment (Singh Karam *et al.*, 2022). Rice husk biochar has shown notable potential for controlled P release, as its porous structure and surface functionalities facilitate P adsorption and gradual nutrient delivery (Ramesh & Raghavan, 2024; Willian da Silva *et al.*, 2024). Impregnation with a phosphate solution is one strategy to enhance P retention. The efficiency of this process depends on key operational parameters, such as stirring speed, which influences hydrodynamics and external mass transfer, while contact time governs adsorption equilibrium and interaction with functional groups and mineral surfaces (Das & Ghosh, 2023; Parimelazhagan *et al.*, 2022).

Despite extensive research on biochar modification, the combined effects of stirring speed and contact time on phosphate impregnation in rice husk biochar remain understudied. Understanding this mechanism is critical for optimizing biochar-based P fertilizers. Thus, this study investigated the effects of stirring speed and impregnation duration on phosphate impregnation and the

physicochemical properties of rice husk biochar, with particular emphasis on water- and acid- soluble P contents, proximate properties, and mass transfer behavior.

2. Methodology

2.1 Materials

Rice husk was sourced from Belung Village in Bantur District, Malang, East Java, Indonesia. Monoammonium phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$, MAP) was used as the phosphate source, and vanadate-molybdate (vanadate and molybdate ions) reagent was used for phosphate determination. All chemicals were analytical grade.

2.2 Biochar Production

Biochar was produced at the Corporate Laboratory, Faculty of Agricultural Technology, PT Pupuk Indonesia, Universitas Brawijaya. A total of 500 g of rice husk was pyrolyzed at 600 °C using a vertical pyrolysis unit for 2 h. The pyrolysis unit, manufactured by the Faculty of Engineering workshop at Universitas Brawijaya, was equipped with a 3500-watt heating source and automatic temperature control up to 1200 °C. Following cooling to ambient temperature, the biochar was milled in a planetary ball mill (CY-MSK-SFM-1S, Zhengzhou CY Scientific, China) at 100 rpm for 2 h. The milled biochar was then sieved using a Dahan DH-300T with a 300-mesh screen (GB/T6003.1-2022, China) to obtain a uniform particle size.

2.3 Impregnation Procedure

MAP was pulverized using a mortar and pestle prior to impregnation. The ground phosphate was combined with distilled water at a ratio of 3:1 (water:phosphate, w/w) and agitated for 15 minutes at 400 rpm. Biochar was then incorporated into the solution to reach a final proportion of 3:1:1 (water:phosphate:biochar, w/w/w). The mixture was stirred for 15 minutes at either 600 or 1200 rpm, followed by incubation for 6, 12, 18, 24, or 30 h. At the end of each incubation period, the solid and liquid phases were separated using Whatman Grade 42 ashless quantitative filter paper (Cytiva; nominal particle retention 2.5 µm). The filtrate was collected, and its recovered volume

was recorded. Finally, the impregnated biochar was oven-dried (UN55, Memmert, Germany) at 105 °C for 24 h. Each combination of stirring speed and impregnation duration was performed using three experimental replicates ($n = 3$). Each replicate consisted of a separately prepared impregnation experiment. Analytical measurements were performed on each experimental unit, and the results are reported as mean $\pm$ standard deviation.

2.4 Proximate Analysis

Moisture, ash, and volatile matter contents were quantified based on the mass losses obtained after heating the samples for 3 h at 115 ± 5 °C (International Organization for Standardization, 2024), 2 h at 800 °C, and 7 minutes at 900 °C, respectively, in a muffle furnace (Linberg/Blue M, Thermo Fisher Scientific, US), following procedures adapted from ASTM guidelines (ASTM International, 2021). The fixed carbon (FC, %) was calculated as the residual mass fraction after deducting the measured percentages of volatile matter, ash, and moisture from the total mass (Equation 1).

$$FC (\%) = 100\% - (\text{Moisture Content } (\%) + \text{Ash Content } (\%) + \text{VM } (\%)) \quad (1)$$

Where FC is fixed carbon (%) and VM is volatile matter (%)

2.5 Water- and Acid-soluble P content

Water- and acid-soluble P were determined using a UV-Vis spectrophotometer (UV-1780, Shimadzu, Japan) following the vanadate-molybdate colorimetric method. Absorbance was measured at 490 nm, and P concentration was converted to elemental P content based on extraction volume, dilution factor, sample mass, and dry matter correction. The results are reported as elemental P on a dry-mass basis (% w/w).

2.6 Mass Transfer Analysis

Mass transfer during phosphate impregnation was evaluated using a pseudo-first-order model commonly applied to solid-liquid systems under well-mixed conditions. The phosphate concentration in the liquid phase at each sampling time was estimated by mass balance from the measured phosphate uptake in the biochar using Equations 2 and 3:

$$\ln \left(\frac{c_0}{c(t)} \right) = K_L a t \quad (2)$$

where:

$K_L a$ t: Volumetric mass transfer coefficient (min^{-1})

C_0 : initial phosphate concentration (mg/L)

$C(t)$: phosphate concentration at a given time (mg/L)

t : impregnation time (min)

The instantaneous mass transfer rate was calculated as follows:

$$N_A(t) = K_L a C(t) \quad (3)$$

where $NA(t)$ ($\text{mg L}^{-1} \text{min}^{-1}$) represents the phosphate transfer rate from the liquid phase to the biochar at time t . As $C(t)$ decreases with time, $NA(t)$ likewise declines, reflecting reduced driving force and progressive saturation of adsorption sites.

2.7 Statistical Analysis

R software version 4.5.1 and the RStudio environment for Windows were used to conduct the statistical analysis (R Core Team, 2025). Descriptive statistics and two-way ANOVA were performed using the `aov()` function from the `stats` package and the `car` package to assess significant differences among biochar samples following impregnation. When significant effects were identified, treatment means were compared using Tukey's post hoc test at a 95% confidence level.

3. Results and Discussion

3.1 Rice Husk Biochar Characteristics

A compositional analysis of rice husks and the resulting biochar was conducted to evaluate changes in moisture, ash, volatile matter, and fixed carbon following pyrolysis. These properties reflect the physicochemical transformations that occur during thermal conversion and determine the suitability of biochar for soil improvement and carbon storage applications (Lehmann & Joseph, 2015).

Table 1. Rice husk and rice husk biochar characteristics

Material	Moisture (%)	Ash (%)	Volatile matter (%)	Fixed Carbon (%)
Rice husk	9.10 ± 0.05 ^a	29.00 ± 0.24 ^a	32.11 ± 2.22 ^a	29.78 ± 2.25 ^a
Rice husk biochar	4.81 ± 0.60 ^b	17.67 ± 0.49 ^b	19.00 ± 0.00 ^b	58.52 ± 0.56 ^b

Different lowercase letters in the same column indicate significant differences between the materials for each parameter.

Pyrolysis markedly reduced the moisture content ($p = 0.000553$), reflecting the typical dehydration and structural stabilization that occur during thermal conversion. Lower moisture content is advantageous for stabilizing pore development and improving subsequent modification or impregnation processes, as excessive water can impede reagent diffusion and limit uniform nutrient uptake (James R. *et al.*, 2020).

Ash content significantly decreased after pyrolysis ($p = 8.114\text{e-}06$), despite the common expectation of ash enrichment due to organic mass loss. This reduction can be attributed to specific process conditions, such as high temperatures, fast pyrolysis, or volatilization of certain minerals (Puri *et al.*, 2024). This underscores the importance of understanding both the ash composition and process parameters in interpreting biochar properties.

Volatile matter (VM) reflects the thermal behavior of biomass during the pyrolysis process. Our data demonstrated that VM markedly declined in biochar ($p = 0.001116$). This trend aligns with findings from goldenrod (Łapczyńska-Kordon *et al.*, 2022) and cotton stalk (Chen *et al.*, 2015), where higher pyrolysis temperatures accelerated devolatilization (Claoston *et al.*, 2014). The loss of VM reflects the breakdown of hydrophilic groups and organic compounds (Zhou *et al.*, 2012), and the release of volatiles can generate new porosity that increases surface area and adsorption potential (Lita *et al.*, 2022).

The fixed carbon of rice husk biochar nearly doubled compared to that of raw rice husk ($p = 6.18\text{e-}05$), indicating enhanced structural carbon stability. A high fixed carbon level is associated with greater resistance to microbial degradation, supporting long-term carbon sequestration and soil fertility benefits through nutrient retention and slow carbon release (Fernandes *et al.*, 2022; Kumar *et al.*, 2024).

3.2 Moisture Content of Phosphate-Impregnated Rice Husk Biochar

Moisture content declined progressively with longer impregnation at both stirring speeds, indicating that contact time predominantly governs water

desorption from the biochar matrix (Figure 1). Statistical analysis confirmed significant effects of stirring speed, impregnation time, and their interaction on moisture content, with impregnation time showing the strongest effect ($p = 2.905\text{e-}12$), followed by stirring speed ($p = 3.398\text{e-}05$), and the speed $\times$ time interaction ($p = 0.0207$). The Tukey post hoc test identified 600 rpm for 6 h as the highest-moisture group (f) and 1200 rpm for 30 h as the lowest (a), while overlapping groups at 24–30 h suggest that longer impregnation reduced the speed-related differences in moisture content. Compared to untreated biochar (Table 1), all impregnated biochar consistently exhibited lower moisture levels. Moisture content is closely linked to the pore architecture and surface area of biochar, consistent with Shomana *et al.* (2020), who reported that sunflower husks retain more moisture than wood chips due to differences in pore size distribution, which affects the hydrophilic character of the biochar.

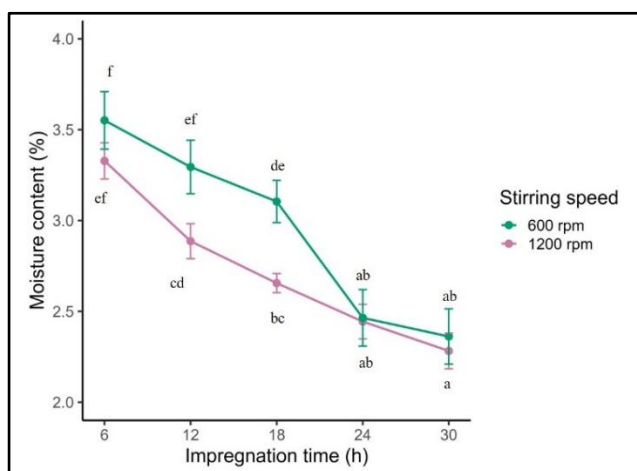


Figure 1. The moisture content (%) of phosphate-impregnated rice husk biochar following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

Although impregnation time was the dominant factor affecting the moisture content, stirring speed also contributed to the reduction of moisture content. Higher stirring speed contributed to a lower moisture content by enhancing the penetration of the MAP solution and promoting the release of water-bound ions from the biochar matrix through increased hydrodynamic forces (He *et al.*, 2023). Longer impregnation time further enhanced the interaction of MAP solution ions, such as NH_4^+ and H_2PO_4^- , with biochar pores, increasing water desorption and leading to greater water evaporation during the drying phase. Although hydrophilic functional groups and the hygroscopic nature of MAP

may initially retain moisture, impregnation can modify the biochar surface to become more hydrophobic, facilitating water release (Iskandar & Rofiatin, 2017). Moisture content also influences the functionalization efficiency because high moisture content hinders impregnant diffusion (Peter *et al.*, 2021).

3.3 Ash Content of Phosphate-Impregnated Rice Husk Biochar

Ash content reflects the thermally stable inorganic elements that remain after combustion. In biochar, high ash content is often correlated with increased levels of key nutrients, including potassium, calcium, and magnesium, that enhance soil properties (Domingues *et al.*, 2017; Grafmüller *et al.*, 2022). This study analyzed the ash content of phosphate-impregnated rice husk biochar to assess the impact of impregnation conditions.

As shown in Figure 2, ash content was significantly affected by stirring speed ($p = 0.0131$) and impregnation duration ($p = 1.230e-10$), while their interaction was not significant ($p = 0.5134$), with impregnation duration exerting a dominant effect. The Tukey post hoc test showed that the lowest ash content occurred at 600 rpm for 6 h (a), whereas 1200 rpm for 24 h, 600 rpm for 30 h, and 1200 rpm for 30 h were grouped as the highest ash treatments (e). Ash content increased progressively with longer impregnation times and higher stirring speeds, reaching $16.78 \pm 0.31\%$ at 600 rpm and $16.96 \pm 0.55\%$ at 1200 rpm for 30 h. This trend confirms that mineral enrichment occurred primarily as a function of extended MAP and biochar interactions. Although stirring speed showed a statistically significant effect, its contribution was relatively small compared with that of impregnation duration. The results suggest that while agitation may support uniform MAP dispersion, impregnation duration is the dominant factor controlling ash accumulation in the treated biochar.

The rise in ash content is primarily attributed to the addition of MAP, which enriches the ash content of P-impregnated biochar. Additional contributions arise from feedstock composition and chemical reactions occurring during impregnation and pyrolysis. The ash content in biochar primarily consists of inorganic minerals that remain after the combustion of organic material. Impregnation and thermal treatment often elevate the ash content by enhancing mineral retention. For example, the impregnation of olive mill waste-derived biochar with olive mill wastewater resulted in increased mineral content by 12%, reflecting added minerals such as sodium, phosphorus,

potassium, calcium, and iron (El-Bassi *et al.*, 2021). This supports the finding that mineral enrichment during pyrolysis promotes the formation of inorganic residues, leading to elevated ash levels in the final product (Oyeyiola, 2021).

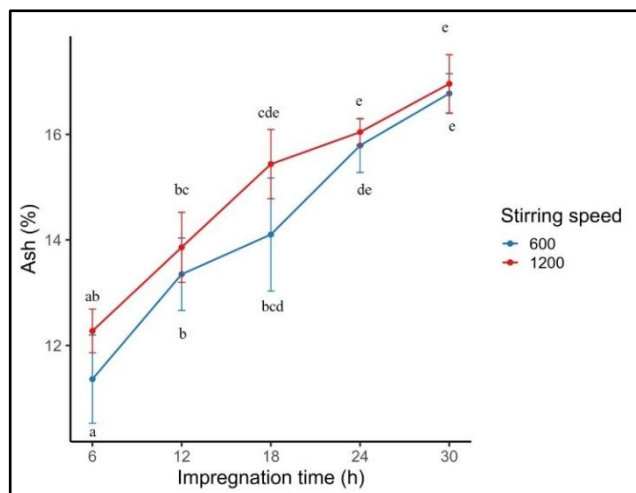


Figure 2. The ash content (%) of phosphate-impregnated rice husk biochar following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

3.4 Volatile Matters of Phosphate-Impregnated Rice Husk Biochar

The volatile matter (VM) gradually decreased with increasing stirring speed and impregnation time, as shown in Figure 3. The reduction was mainly driven by impregnation duration, with VM decreasing consistently from 6 to 30 hours. Statistical analysis showed that stirring speed ($p = 8.229\text{e-}04$) and impregnation time ($p = 1.778\text{e-}12$) significantly affected the volatile matter, while their interaction was not significant ($p = 0.6331$); the overall pattern indicated that time is the primary determinant of VM loss. The Tukey post hoc test identified 600 rpm for 6 h as the highest VM (e), whereas 1200 rpm for 30 h, 600 rpm for 30 h, and 1200 rpm for 24 h were grouped as the lowest treatments (a). This trend aligns with that of Li *et al.* (2020), who reported that phosphate addition promotes dehydration and breakdown of labile carbon structures during thermal processing, thereby reducing volatile carbon in biochar. Prolonged contact between biochar and phosphate solution likely enhanced phosphate penetration and interaction, accelerating the conversion of volatile components into more stable carbon forms.

Although the variation in stirring speed was small, the 1200 rpm treatment consistently demonstrated a slightly lower VM content than 600 rpm treatment, indicating that increasing the stirring speed may improve VM release. Higher stirring speed reportedly enhanced mass transfer of the impregnant into the biochar's porous structure, promoting more uniform interaction and efficient permeation. Increased stirring also improves the contact between biochar and the impregnation agent, thereby accelerating the reaction kinetics (Kavand & Kazemian, 2025; Sutar & Jadhav, 2025).

Impregnation compounds, particularly metals or salts, may bind to the surface functional groups on biochar, forming stabilized structures that reduce VM by immobilizing or transforming volatile components (Deveci *et al.*, 2024). Additionally, during impregnation, especially at elevated temperatures, volatile organic compounds, cellulose, and hemicellulose in the biochar decompose. Mechanical agitation accelerates this breakdown, reducing the volatile matter and increasing the biochar stability through higher fixed carbon retention (Fang *et al.*, 2014).

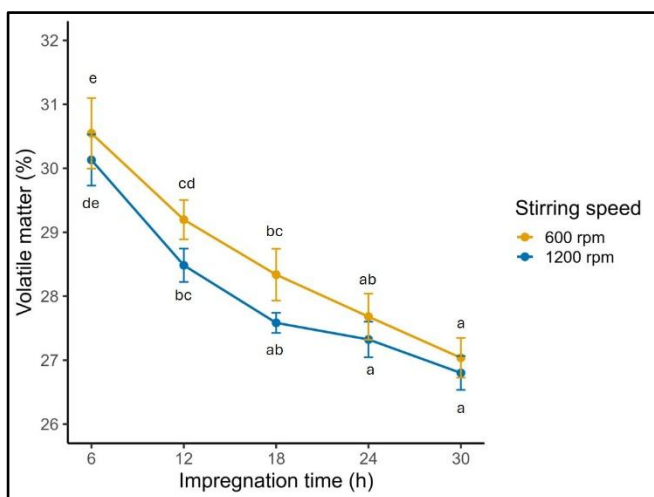


Figure 3. The volatile matter content (%) of phosphate-impregnated rice husk biochar following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

3.5 Fixed Carbon (%) of Phosphate-Impregnated Rice Husk Biochar

The FC content did not differ statistically across different stirring speeds ($p = 0.7406$) and impregnation times ($p = 0.6394$), indicating a minimal impact

from these treatments. However, when compared to untreated biochar (58.52%) as presented in Table 1, all impregnated samples exhibited a lower FC level (Figure 4). As reported in earlier studies, this reduction is mainly linked to the chemical reactions occurring during impregnation, as well as the nature of the impregnation materials and their interaction with the biochar matrix, which can promote the depolymerization of cellulose and lignin, primary carbon sources, thereby reducing the formation of a stable carbon structure (Chang *et al.*, 2025; Murtaza *et al.*, 2024). Thus, impregnation did not significantly alter FC between treatments, but the impregnation process as a whole resulted in a measurable decrease in FC relative to the original biochar.

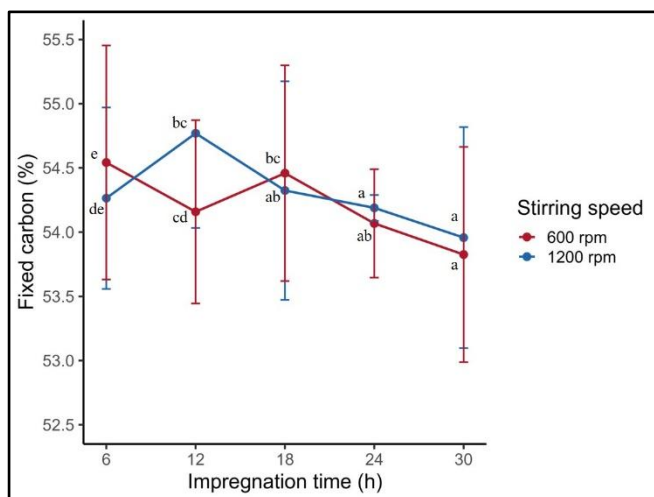


Figure 4. The fixed carbon content (%) of phosphate-impregnated rice husk biochar following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

3.6 Water- and Acid-Soluble P Content of Phosphate-Impregnated Rice Husk Biochar

Following MAP impregnation, the water-soluble P content increased significantly with longer impregnation times ($p = 1.544\text{e-}18$) and higher stirring speeds ($p = 4.445\text{e-}06$), whereas their interaction was not significant ($p = 0.3425$), indicating a generally consistent time-dependent increase across both speeds (Figure 5A). The Tukey post hoc test showed that the lowest values occurred at 600 and 1200 rpm for 6 h (a), whereas the highest value was observed at 1200 rpm for 30 h ($14.350 \pm 0.450\%$, f). However, this

treatment was not significantly different from 600 rpm for 30 h (def) and 1200 rpm for 24 h (ef), indicating that water-soluble P content approached a plateau after prolonged impregnation. This trend reflects the enhanced interaction between MAP and the surface functional groups on biochar over a prolonged contact period, facilitating phosphate complex formation and deeper ion diffusion into the porous biochar structure, thus enhancing phosphate retention and solubility (Wang *et al.*, 2022). Higher stirring speeds further improve mass transfer, reduce concentration gradients, and promote uniform MAP dispersion across the biochar matrix (Gajendiran *et al.*, 2025). MAP hydrolysis in aqueous solutions generates available phosphate ions, and both the duration and stirring support more effective hydrolysis and uptake (Qiu *et al.*, 2023).

A similar pattern was observed for acid-soluble P, where stirring speed ($p = 4.868\text{e-}06$) and impregnation time ($p = 4.020\text{e-}21$) significantly affected acid-soluble P content, while their interaction was not significant ($p = 0.2487$), indicating a consistent time-dependent increase across both stirring speeds (Figure 5B). The Tukey post hoc test showed that the lowest acid-soluble P was obtained at 600 rpm for 6 h (a), while the highest value was recorded at 1200 rpm for 30 h ($15.787 \pm 0.186\%$, g). This treatment was not significantly different from 600 rpm for 30 h (fg), suggesting that prolonged impregnation was the main factor driving acid-extractable P accumulation. Enhanced agitation and prolonged impregnation promote deeper penetration of phosphate and bonding with the functional groups present on the biochar surface, such as carboxylic and hydroxyl groups, forming more acid-soluble complexes (Jung *et al.*, 2016; Zhao *et al.*, 2016). Citric acid extraction creates an acidic environment, and the higher recovery in this fraction indicates greater phosphate extractability under elevated proton activity, which facilitates dissolution and desorption of phosphate species (Morales *et al.*, 2013; Niedzbała *et al.*, 2024; Uchimiya & Bannon, 2013).

Furthermore, changes in phosphate speciation under acidic conditions may also enhance solubility, as H_2PO_4^- dominates at low pH and is more readily mobilized (Antunes *et al.*, 2018; Niedzbała *et al.*, 2024). Additionally, organic acids can facilitate the release of phosphate bound to carboxyl-rich surfaces (Kharel *et al.*, 2019), contributing to the increased extractability.

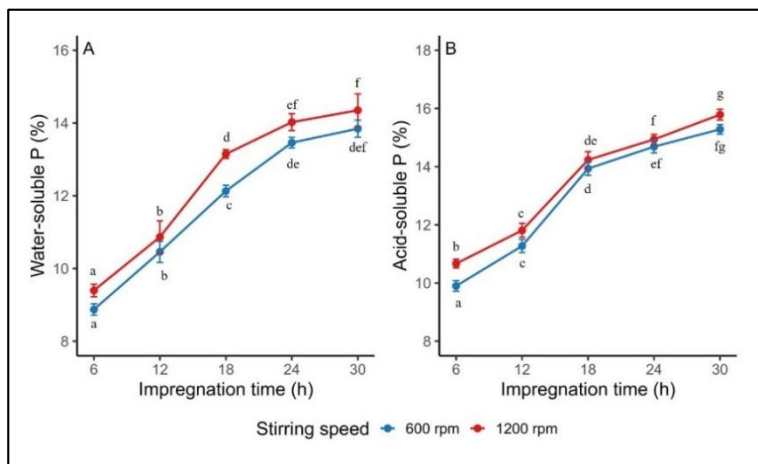


Figure 5. The water-soluble (A) and acid-soluble (B) P content (%) of phosphate-impregnated rice husk biochar following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

3.7 Mass Transfer Analysis

The mass transfer of phosphate from MAP into rice husk biochar was evaluated for both water-soluble and acid-soluble fractions across impregnation times and stirring speeds (Figure 6A–B). In the present study, the water-soluble fraction provides an operational measure of phosphate that is readily extractable in water, whereas the acid-soluble fraction provides an operational measure of phosphate extractable under acidic conditions. Therefore, the acid-soluble fraction may represent phosphate that is relatively less immediately water-soluble; however, it should not be interpreted as direct evidence of slow-release behavior. These measurements were used to evaluate the influence of impregnation conditions on phosphate incorporation and mass transfer behavior.

Mass transfer rates calculated from both P fractions declined sharply from 6 to 30 hours. For acid-soluble P fraction, rates decreased from $1.28 \text{ mg L}^{-1}\cdot\text{h}^{-1}$ (1200 rpm) and $1.23 \text{ mg L}^{-1}\cdot\text{h}^{-1}$ (600 rpm) at 6 hours to approximately 0.31 and $0.29 \text{ mg L}^{-1}\cdot\text{h}^{-1}$ at 30 hours. Water-soluble P fraction exhibited comparable behavior, dropping from 1.26 to $0.30 \text{ mg L}^{-1}\cdot\text{h}^{-1}$ (1200 rpm) and from 1.20 to $0.30 \text{ mg L}^{-1}\cdot\text{h}^{-1}$ (600 rpm). The slightly higher initial values at 1200 rpm suggest improved hydrodynamic conditions that enhance early-stage diffusion, although this effect diminishes as the system approaches equilibrium.

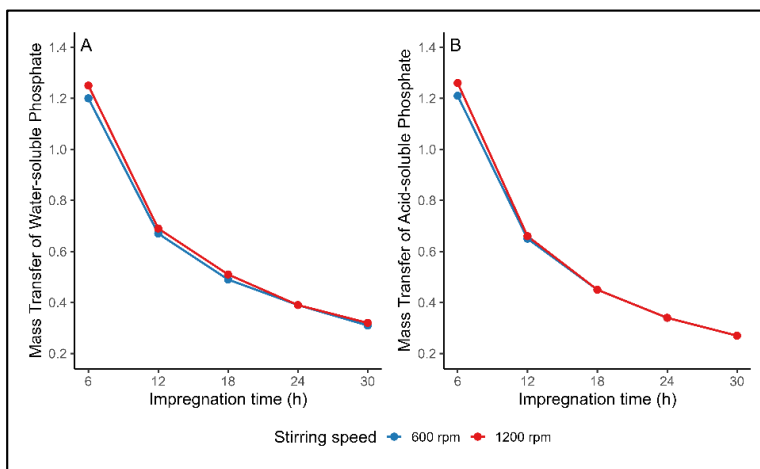


Figure 6. The mass transfer rate of water-soluble (A) and acid-soluble (B) P following various impregnation times (6, 12, 18, 24, and 30 hours) and stirring speeds (600 and 1200 rpm)

The declining trend primarily reflects the progressive saturation of the adsorption sites. During the first hours, abundant functional groups and pore structures allow rapid phosphate uptake; as these sites become occupied and the solution concentration decreases, the driving force for mass transfer weakens. Such behavior corresponds with the characteristic drop in adsorption rate once readily accessible sites are filled (Tu *et al.*, 2024). Structural changes within the biochar may further contribute to reduced rates at longer durations. Impregnation can partially block pores, limiting access to the internal surface and slowing diffusion, similar to observations where metal loading reduces porosity and mass transport efficiency (Micháleková-Richveisová *et al.*, 2017).

Additionally, at longer durations, the interactions between phosphate ions and biochar may also be influenced by competition from other ions present in the solution. As the concentration of phosphate declines, other anions present may interact with the binding sites, further reducing the effectiveness of phosphate adsorption. Studies have highlighted how additional anions can inhibit phosphate uptake through competition for adsorption sites, thereby exacerbating the decline in transfer rates during impregnation (He *et al.*, 2024; Li *et al.*, 2016). However, once the system nears saturation, further increases in agitation have a limited effect because the concentration gradient and the associated driving force are substantially diminished (Zhang *et al.*, 2022).

4. Conclusions and Recommendations

This study shows that impregnation duration is the primary factor influencing phosphate impregnation and the physicochemical changes in rice husk biochar. Longer contact times consistently reduced moisture and volatile matter, increased ash content, and enhanced both water- and acid-soluble P contents. Stirring speed had only a modest effect, improving early-stage mixing but not substantially altering outcomes at extended durations. Mass transfer rates were highest during the first 6 h and declined as adsorption sites became saturated and diffusion resistance increased. Within the investigated range, the highest phosphate contents were observed after 30 h of impregnation. However, some of the 30 h treatments were not significantly different from the 24 h treatments. Therefore, the results support a dominant effect of impregnation duration rather than the identification of a unique optimal treatment.

Since the present study did not evaluate phosphate release kinetics under soil or aqueous conditions, the water- and acid-extractable P fraction should not be interpreted as direct evidence of slow-release fertilizer performance. These fractions describe phosphate extractability under the respective extraction conditions. Further studies integrating structural characterization, soil incubation, nutrient release kinetics, and plant uptake are required to evaluate the potential of phosphate-impregnated biochar as a slow-release fertilizer.

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