

# Kinetic Release Models of Nitrogen and Iron from Chitosan-Biochar Based Slow Release Fertilizers

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## Abstract

The use of slow-release fertilizers (SRFs) is increasingly recognized as a sustainable method to enhance nutrient-use efficiency. This study utilized nitrogen from urea and Fe from iron sand, both encapsulated in chitosan–biochar matrices through NaOH-induced solidification to form bead-type SRFs. Four formulations with different biochar–urea ratios were prepared: A (CB2U2Fe), B (CB3U2Fe), C (CB3U3Fe), and D (CB2U3Fe). Bead morphology was examined using scanning electron microscopy (SEM), while nitrogen and Fe release behavior was evaluated after 1, 3, 5, and 7 days under soil percolation. Release patterns were modeled using pseudo-first-order (PFO), pseudo-second-order (PSO), Weber–Morris, and Elovich kinetics. SEM results showed that chitosan–biochar–urea beads (BCB@N) contained numerous voids and surface cracks, indicating a porous microstructure. In contrast, chitosan–biochar–urea–Fe beads (BCB@UFe) displayed a denser structure with fewer pores, supporting more controlled water entry and nutrient diffusion. Release experiments identified sample C as the most stable formulation for both nitrogen and Fe. Nitrogen-release kinetics fitted the PFO ( $R^2 = 0.920$ ) and PSO ( $R^2 = 0.954$ ) models, indicating diffusion-driven release with weak surface interactions. Fe-release kinetics for sample C aligned best with the PFO and Weber–Morris models, suggesting dominance of intra-particle diffusion and physisorption processes.

**Keywords:** biochar-chitosan beads; slow release; Nitrogen; Fe; release kinetic models.

## INTRODUCTION

The use of conventional fertilizers is being gradually replaced as modern agriculture is rapidly implemented, taking into account the negative environmental impacts of agricultural activities. One innovative approach developed to support modern agriculture is the use of slow-release fertilizers (SRF). This fertilizer is designed to minimize the environmental impact of nutrient leaching, which contributes to water pollution, improve fertilization efficiency, and reduce evaporation rates that contribute to increased greenhouse gas emissions. SRF is a method designed to produce fertilizers that release nutrients in a controlled manner according to plant needs, directly supporting sustainable agriculture (Karbeka, 2025; Shaviv, 2001).

SRF innovations are developed using synthetic polymer or organic-inorganic hybrid materials to obtain a coating matrix that can control the rate of nutrient release. Several researchers have demonstrated that synthetic polymer matrices offer mechanical strength and water resistance, as seen in urea-polyacrylamide (PAM). In contrast, the use of natural polymers, such as chitosan, zeolite, bentonite, biochar, and silica, affects the increase in porosity, resulting in a slowdown in nutrient release

due to multiporosity diffusion (Martasiana Karbeka, Zakarias Mautuka, 2024; Sandeep Jakkula & Wani, 2018; Zhou et al., 2018).

Several studies have demonstrated that polymer-based and inorganic-organic hybrid beads possess significant potential as nutrient delivery systems, owing to their stable, porous nature and ability to encapsulate multiple types of nutrients (Manzoor et al., 2022; Sholeha et al., 2024). The performance of slow-release fertilizers is greatly influenced by the composition of the constituent materials, microstructure, and nutrient diffusion mechanism within the bead matrix. Therefore, understanding the kinetics of nutrient release is crucial for predicting nutrient availability to plants and optimizing efficient fertilizer formulations (Danarto et al., 2017; Lakshani et al., 2023). The morphological characteristics and microstructure of SRF components play a crucial role in determining their nutrient release behavior. Analysis using scanning electron microscopy (SEM) allows detailed observation of the surface, porosity, and particle distribution in the bead matrix, which directly affects the mechanism and rate of nutrient diffusion. Structures with uniform pores and good interparticle connectivity tend to result in more controlled nutrient release, while non-homogeneous

structures can accelerate release due to uneven diffusion (Herlina et al., 2025; Sojan et al., 2025). Therefore, morphological studies using SEM are highly relevant for integration with release kinetics analysis to provide a comprehensive understanding of the behavior of slow-release fertilizers. Chitosan and biochar matrix hybrids have been extensively studied, particularly for the release of nitrogen nutrients. The use of biochar also provides long-term benefits as a soil conditioner (Finalis et al., 2020; Griselda et al., 2024; Martasiana Karbeka, Zakarias Mautua, 2024; Martasiana Karbeka, 2022).

Chitosan, as a natural polymer, plays a role in regulating the rate of nutrient release, while biochar, as a porous material, absorbs or temporarily stores nutrients. The use of porous materials enables the storage of various types of nutrients (Fatma Nur Parin, Kenan Yildirim, 2020; Yu et al., 2024). Nutrient release kinetics analysis is a key approach in evaluating the efficiency of SRF systems. Pseudo-first-order (PFO) and pseudo-second-order (PSO) models are used to describe release controlled by physical or chemical interactions. The Weber–Morris model explains the role of intraparticle diffusion, and the Elovich model is used for systems with heterogeneous surfaces (Karbeka, 2024; Shafiq et al., 2021). The combination of SEM data and kinetic analysis provides a deeper understanding of the structure-function relationship, particularly for the release of nitrogen (N) and iron (Fe) from SRF bead-based systems. Use 170 x 250 mm paper size (W x H mm) and adjust the margins to those shown in the Table 1. The final printed area will be 130 x 210 mm. Do not add any page numbers.

## MATERIALS AND METHODS

### Materials

Materials used in this study were biochar, chitosan, urea (source nitrogen), iron sand (source Fe), NaOH (Merck), magnetic stirrer, chemistry glassware, analytical balance and stirring rod, indicator PP, scanning electron microscopy (SEM), atomic absorption spectroscopy (AAS).

### Procedures

#### Synthesis and SEM Characterization

SRF beads were synthesized in four variations  $CB_2U_2Fe$ ;  $CB_3U_2Fe$ ;  $CB_3U_3Fe$  and  $CB_2U_3Fe$ . Sample description

as follows C (chitosan); B (biochar); U (urea). Urea, a source of nitrogen, was dissolved, then mixed with biochar, and subsequently added to the chitosan solution. The mixture was stirred using a magnetic stirrer until it became homogeneous. Iron sourced from iron sand was added and stirred manually without a magnetic stirrer until the mixture was evenly blended. The resulting mixture was dripped into 2 M NaOH using a syringe, and each drop solidified in the NaOH solution to form alkaline beads. The resulting beads were tested for neutralization. Since the beads were alkaline, a neutralization process was carried out through washing, followed by a negative test using phenolphthalein (PP) indicator. The beads obtained were characterized using SEM for two types of bead samples, namely beads containing BCB@N nitrogen and beads containing nitrogen and Fe (BCB@NFe).

#### Nitrogen and Fe Release Test

Each bead composition was mixed with soil using the percolation method to simulate the release of nutrients in soil exposed to water flow. Water was poured at a constant volume (500 mL) for a week, and the resulting leachate was collected in separate containers for each day of watering. All leachate samples obtained were collected on days 1, 3, 5, and 7 for analysis of nitrogen and Fe release. Each water sample was filtered to remove solid particles, and the amount of nitrogen released was analyzed using the Kjeldahl method, and Fe release was analyzed using atomic absorption spectroscopy (AAS).

## RESULTS AND DISCUSSION

### SEM Characterization

This study successfully synthesized four variations of beads, along with nitrogen-loaded beads (BCB@N) and nitrogen–Fe loaded beads (BCB@NFe), which were produced through the base gelation method with NaOH as the precipitating agent. The internal morphology of the beads was examined through a comparative analysis between (BCB@N) and (BCB@NFe), aiming to identify the general effects of N and N–Fe incorporation on their internal structure. The SEM images are presented in Figure 2.

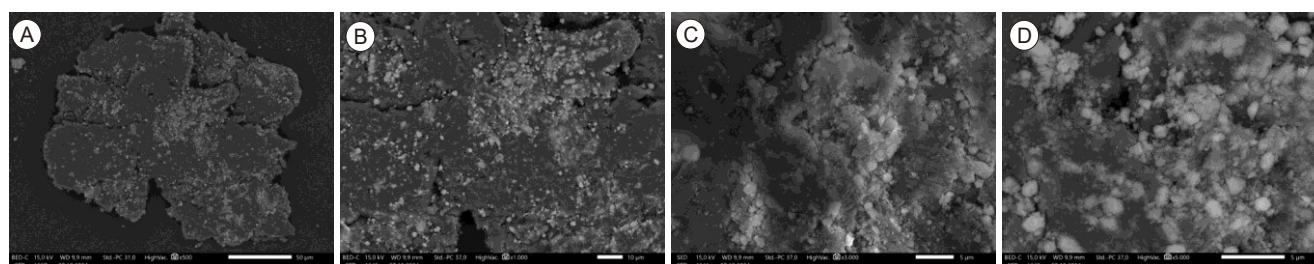


Figure 1. SEM image of BCB@N (a)500x;(b)1000x;(c)3000x;(d) 5000x.

The surface morphology of BCB@N shows a heterogeneous structure. A magnification of 500x reveals an irregular surface, indicating an uneven distribution of the biochar chitosan composite matrix. The rough, lumpy surface is characteristic of the biopolymer layer. At 1000x magnification, biochar aggregation within the chitosan polymer is visible, forming micropores and mesopores. The formation of porosity plays a crucial role in the process of water penetration and nutrient release. The presence of a layer creates a diffusion barrier,

thereby controlling the release of nutrients (Herlina et al., 2025). High magnification at 3000x and 5000x shows cracks, voids, and a rough surface. The microstructural defects are not interconnected, allowing the layer structure to resist the rate of nitrogen dissolution because the voids and cracks act as pathways for water penetration during nitrogen diffusion. The isolated pores and cracks in BCB@N can prevent excessive nitrogen leaching, resulting in more controlled release.

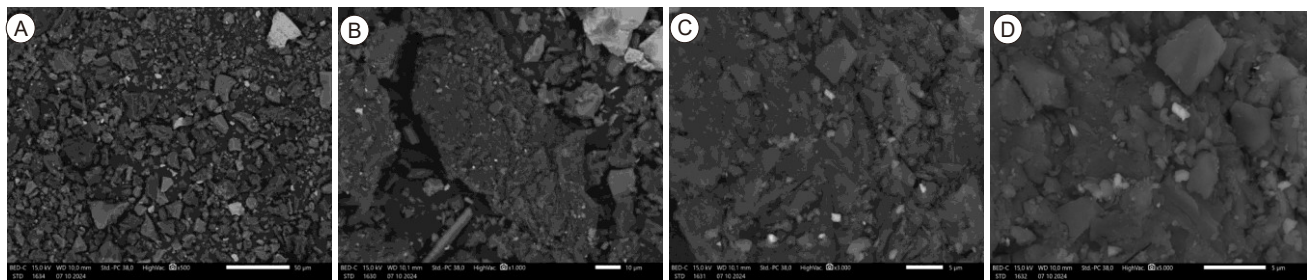


Figure 2. SEM image of BCB@NFe (a) 500x; (b) 1000x; (c) 3000x; (d) 5000x.

The surface morphology of BCB@NFe shows a layered and non-homogeneous structure. The dense surface appearance with fine particles scattered throughout indicates the successful aggregation of Fe from iron sand and urea, which is coated with a chitosan-biochar matrix. Observation at 1000x magnification reveals the bond between urea and Fe, with small gaps forming in the chitosan biochar network system. The presence of cracks and voids in the microstructure provides a pathway for water to penetrate the layer that protects the nutrients, thereby slowing down the diffusion of nutrients. The bright solid surface is a deposit of Fe oxide distributed on the surface of BCB@NFe and strengthens the mechanical properties of the coating matrix. At magnifications of 3000x and 5000x, the surface appears rough, with nano- and micrometer-sized particles distributed throughout (14). The aggregation of urea and Fe particles coated by the chitosan-biochar matrix is clearly visible

### Release of Nitrogen from Slow-Release Fertilizer Beads

Nitrogen (N) release kinetics from four variations of slow-release fertilizer beads based on chitosan–biochar–urea, based on the coefficient of determination ( $R^2$ ) values for four kinetic models, namely pseudo-first order (PFO), pseudo-second order (PSO), intra-particle diffusion (Weber–Morris), and Elovich.

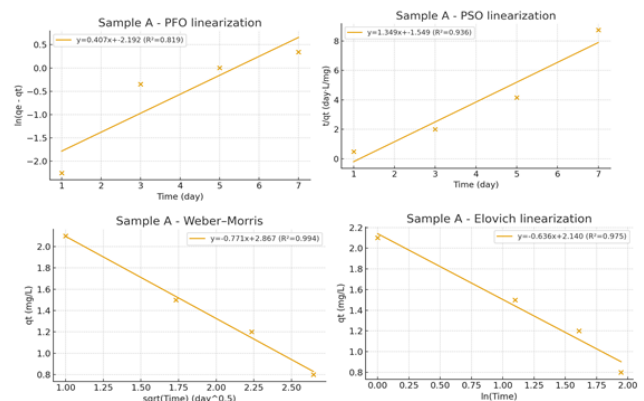


Figure 3. Kinetic model of N release, sample A (CB2U2Fe).

In sample A, there was a correlation in nitrogen release based on the highest  $R^2$  values obtained in the Weber–Morris ( $R^2 = 0.994$ ) and Elovich ( $R^2 = 0.975$ ) kinetic models, followed by PSO ( $R^2 = 0.936$ ). This indicates that nitrogen release in sample A beads is strongly influenced by the intra-particle diffusion mechanism, which describes the process of nitrogen release from the interior of the matrix to the surface before dissolving into the aqueous medium. After the initial surface stage, nitrogen from the interior of the beads moves through the internal pores of the matrix. Due to the small size and weak charge of the molecules, diffusion becomes the main limiting stage. The Weber–Morris model describes this process. The fit to the Elovich model also indicates that the surface of the beads is heterogeneous, allowing adsorption and desorption to occur at different active sites. The relatively high PSO model reinforces that, in addition to diffusion, there is a contribution from weak chemisorption reactions between N and the active groups of chitosan–biochar. Thus,

nitrogen release in sample A beads is controlled by dominant intra-particle diffusion with a contribution from chemisorption.

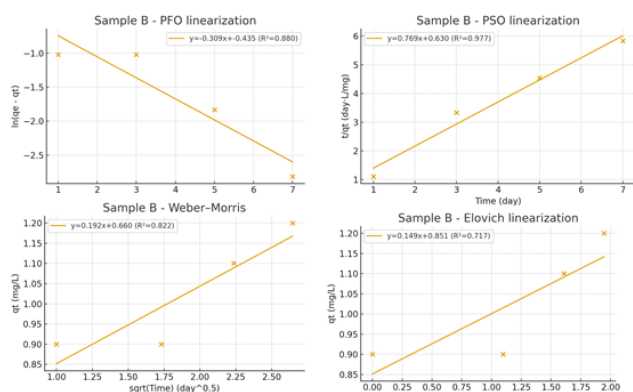


Figure 4. Kinetic model of N release, sample B (CB3U2Fe).

Sample B beads obtained the highest  $R^2$  value for PSO ( $R^2 = 0.977$ ), indicating that the nitrogen release mechanism is dominated by chemisorption (surface reaction between nitrogen and active groups in the matrix) (Karbeka, 2024). Meaning that nitrogen release involves weak interactions between urea/ammonium groups and active groups on the surface of chitosan and biochar, namely amine ( $-NH_2$ ), hydroxyl ( $-OH$ ), and carboxylate ( $-COOH$ ) groups on the surface of chitosan and biochar. These bonds are reversible and weak (hydrogen bonds and van der Waals forces), allowing nitrogen to be easily released back into the water medium.

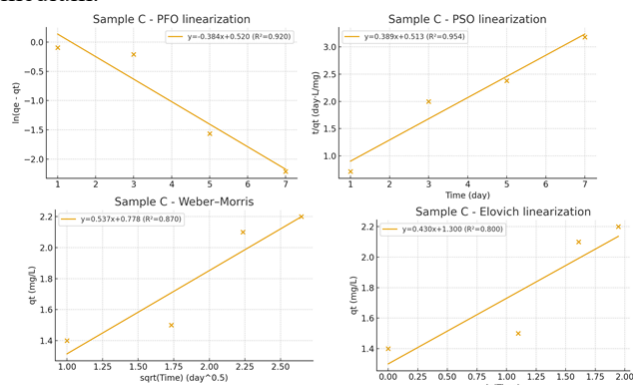


Figure 5. Kinetic model of N release, sample C (CB3U3Fe).

In sample C, the PFO ( $R^2 = 0.920$ ) and PSO ( $R^2 = 0.954$ ) models showed high conformity, indicating that nitrogen release from bead C occurred through a mixed mechanism of physisorption (PFO) and chemisorption (PSO). The combination of high and balanced  $R^2$  values indicates that the pore structure of bead C is sufficiently open to allow for a gradual yet stable release of nitrogen. The release mechanism is jointly controlled by concentration gradients, surface reactions, and diffusion, making beads C the most balanced in the N release system. Nitrogen in the urea fertilizer system is distributed in the form of urea ( $CO(NH_2)_2$ ) and its

hydrolysis products, such as  $NH_4^+$  (ammonium) or  $NH_3$  (ammonia). These species are highly soluble in water and have small molecular sizes. When the beads come into contact with water, nitrogen can more easily move through the pores and exit the matrix through molecular diffusion (Liu et al., 2023).

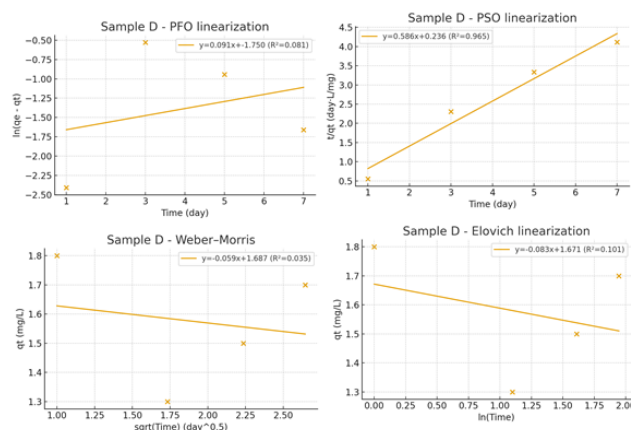


Figure 6. Kinetic model of N release sample D (CB2U3Fe).

In sample D, the  $R^2$  value aligns with the PSO model ( $R^2 = 0.965$ ), indicating a high correlation. This means that nitrogen release is almost entirely controlled by chemisorption reactions specific to the active functional groups on the surface of the beads. The low values of the other models suggest that diffusion processes and surface heterogeneity play a negligible role. This condition may be caused by the beads' structure being too dense or closed, thereby severely limiting N mass transfer through the pores. N release is controlled by a single chemisorption reaction, with the possibility of strong bonds between nitrogen and the matrix functional groups. In general, the PSO model shows the best overall fit (especially for samples B, C, and D), indicating that surface reactions and chemisorption interactions are the primary mechanisms for nitrogen release (Tong et al., 2018). Based on the study of pseudo-first-order (PFO), pseudo-second-order (PSO), intra-particle diffusion (Weber–Morris), and Elovich release patterns, the order of nitrogen release effectiveness and kinetic regularity is sample A < sample B < sample D < sample C, with nitrogen release in sample C (CB3U3Fe) being the most balanced because the nitrogen release process is simultaneously controlled through concentration gradients and weak surface reactions, allowing nitrogen to be released back into the water medium easily.

### Release of Fe from Slow-Release Fertilizer Beads

The kinetic study of Fe release from four variations of chitosan-biochar-urea-based beads was conducted using pseudo-first-order (PFO), pseudo-second-order (PSO), Weber-Morris intra-particle diffusion, and Elovich models. The linearization results for each model are presented in Figure 4, with the coefficient of

determination ( $R^2$ ) values serving as indicators of model suitability for the experimental data.

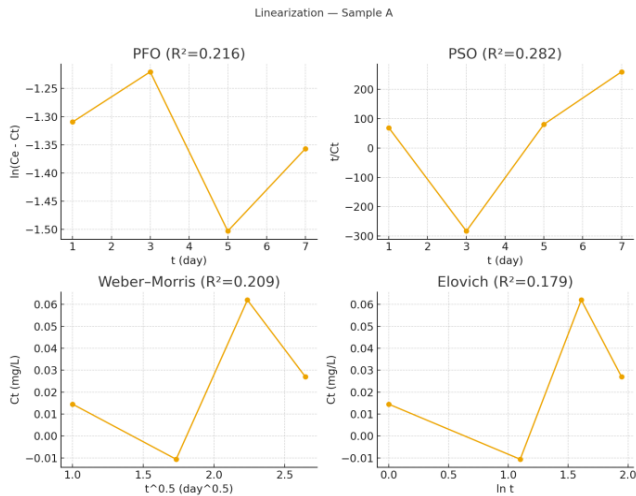


Figure 7. Kinetic model of Fe release, sample A (CB2U2Fe).

In Sample A, the application of the kinetic model used showed low  $R^2$  values (0.179–0.282), which means that Fe release occurred randomly and did not follow any of the kinetic models applied. This pattern indicates that Fe from iron sand has a relatively weak interaction with the chitosan-biochar matrix and may also be due to the unstable structure of the beads, resulting in uncontrolled Fe release through both diffusion and surface adsorption mechanisms.

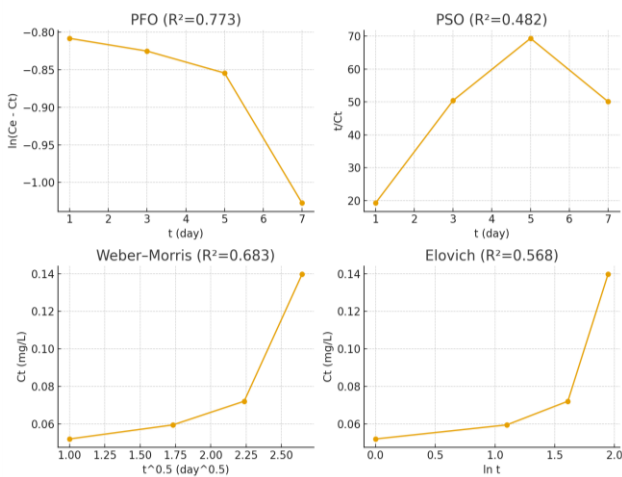


Figure 8. Kinetic model of Fe release, sample B (CB3U2Fe).

Sample B exhibited a distinct pattern, demonstrating the suitability of Fe release in relation to the kinetic models employed, particularly the PFO model ( $R^2 = 0.773$ ) and the Weber–Morris model ( $R^2 = 0.683$ ). Based on the suitability of the PFO model, it can be explained that in sample B, Fe release occurs in a controlled manner due to the concentration gradient between the solution and the surface of the beads. The bead's surface has a higher Fe concentration than the percolate water, causing Fe diffusion and making Fe release a physical

process. The diffusion process also supports the use of chitosan biochar as a porous matrix material in enhancing Fe release through diffusion through the pores of the bead matrix. The combination of PFO and Weber–Morris fits reflects two stages of the Fe release mechanism: (1) an initial rapid stage, where Fe is released from the outer surface through physical desorption, and (2) a subsequent slow stage, which is controlled by the diffusion of Fe from within the pores of chitosan–biochar to the surface. This stepwise release mechanism suggests that the interaction between Fe and chitosan–biochar is weak and concentration gradient-dependent, rather than involving covalent bonding or strong chemical complexation, as commonly described by the PSO model.

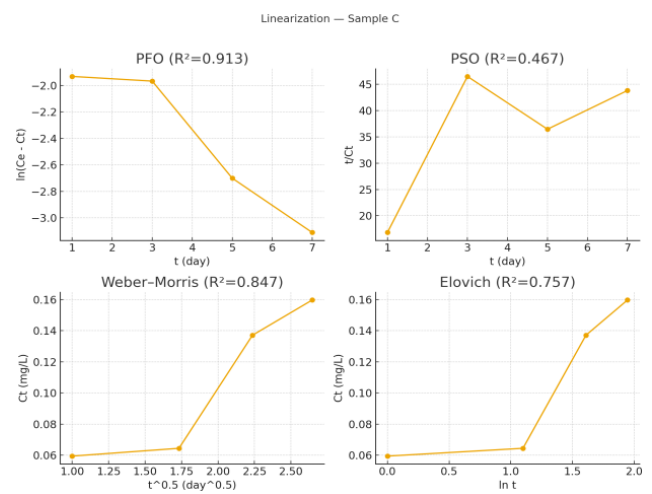


Figure 9. Kinetic model of Fe release, sample C (CB3U3Fe).

In sample C, there is a strong correlation of Fe release following the PFO model ( $R^2 = 0.913$ ), indicating that the rate of Fe release depends on the number of active sites that have not yet released Fe ions on the surface of the coating material. In this system, Fe interacts with amino ( $-\text{NH}_2$ ) and hydroxyl ( $-\text{OH}$ ) groups from chitosan through electrostatic bonds or weak coordination. This type of interaction is reversible and does not form stable chemical complexes, so Fe release occurs through a physisorption–desorption mechanism that can be explained by pseudo-first-order kinetics. This mechanism is consistent with the use of a porous matrix, namely chitosan biochar, which reinforces the suitability of the Fe release pattern. In addition, the Weber–Morris model ( $R^2 = 0.847$ ) confirms that intra-particle diffusion is the rate-limiting step in the release of Fe. This can be explained by the nature of biochar, which has a complex porous structure that allows Fe ions to be trapped in micropores and mesopores during the bead formation process. When the percolate medium enters, Fe must diffuse through the porous network to the surface before being released into the solution

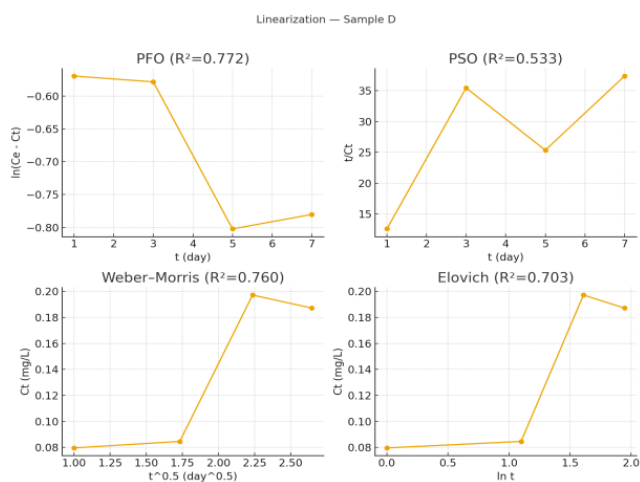


Figure 10. Kinetic model of Fe release, sample D (CB2U3Fe).

For sample D, the release pattern exhibits a level of conformity to the PFO model ( $R^2 = 0.772$ ) and the Weber–Morris model ( $R^2 = 0.760$ ), where Fe tends to undergo physical sorption release, starting with rapid Fe release on the surface and continuing with diffusion within the internal pores. The fit with the Weber–Morris model confirms that intra-particle diffusion is the rate-limiting step for the release of Fe. This can be explained by the nature of biochar, which has a complex porous structure that allows Fe ions to be trapped in the micropores of the matrix during the bead formation process. When the percolate medium enters, Fe must diffuse through the porous network to the surface before being released into the solution. In addition, the Elovich model ( $R^2 = 0.703$ ) also supports the existence of a heterogeneous surface with pore distribution that allows Fe release to occur steadily and gradually, making the release system more controlled.

Based on the release patterns of the four SRF variations, it can be generally concluded that the PFO and Weber–Morris models are the most suitable kinetic models to describe Fe release from the chitosan–biochar–urea-based bead system. The primary release mechanism is governed by intra-particle diffusion and physisorption, rather than chemisorption reactions, as in the PSO model. The order of kinetic suitability efficiency can be described as Sample A < Sample B < Sample D < Sample C, with Sample C showing the most controlled and stable release behavior. This condition can be attributed to a more homogeneous Fe distribution and a more open pore structure in the bead matrix, which facilitates gradual release into the percolation medium.

## CONCLUSIONS

SEM images show that irregular surfaces indicate an uneven distribution of the chitosan biochar composite matrix, and the addition of Fe from iron sand results in a denser surface appearance. Based on the application of kinetic models in determining the release patterns of

nitrogen and Fe in four variations of SRF bead combinations, it is known that controlled and stable Fe release occurs in bead sample C (CB3U3Fe) with  $R^2$  suitability in PFO and moderate  $R^2$  in the Weber–Morris kinetic model. Similarly, for nitrogen release, the kinetic model that showed high conformity with controlled and stable release was found in beads sample C (CB3U3Fe), following the PFO ( $R^2=0.920$ ) and PSO ( $R^2=0.954$ ) models, clearly showing that the chitosan–biochar composition arrangement affects the balance between physical diffusion and chemical surface reactions that determine the rate and stability of nutrient release.

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**Competing Interests:** The authors declare that there are no competing interests.

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