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Eco-Friendly Biochar-Chitosan Coating for Controlled Nitrogen-Fe Release from Slow-Release Fertilizers

Martasiana Karbeka
Department of Chemistry, Faculty of Mathematics and Natural Sciences, Tribuana Kalabahi University, Alor, Indonesia
Jl. Soekarno-Hatta, Batunirwala, Kalabahi-Alor-NTT, Indonesia.

Corresponding author
karbekamartasiana@gmail.com

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Abstract

The development of slow-release fertilizers (SRFs) has gained increasing attention as an efficient approach to enhance nutrient utilization while minimizing nutrient losses and environmental impacts. In this study, biochar–chitosan composites were employed as encapsulating materials for urea and Fe to produce SRF beads. The beads were synthesized through ionic gelation in NaOH solution, followed by swelling tests and structural characterization using Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The swelling analysis demonstrated that the most effective formulation was obtained from the composition containing 3 g of biochar and 3 g of urea, achieving the highest swelling capacity of 92% after 48 h. This result indicates that the biochar–chitosan matrix provides sufficient porosity and water affinity to support nutrient retention and release. FTIR spectra confirmed the presence of characteristic functional groups of biochar, chitosan, urea, and Fe, verifying the successful incorporation of all components into the composite beads. Furthermore, XRD analysis revealed a predominantly amorphous structure, as evidenced by broad peaks in the 2θ range of 20°–60°. However, sharp crystalline peaks were also observed at $2\theta \approx 29.1°$ and 33.9°, corresponding to urea, and at $2\theta \approx 40°$, attributed to crystalline phases of urea and Fe. These results suggest that the structural configuration of the beads combines both amorphous and crystalline phases, which may contribute to controlled nutrient release behavior. Overall, the findings demonstrate that biochar–chitosan encapsulation is a promising strategy for developing efficient SRFs with enhanced swelling properties and structural stability, offering potential for sustainable agricultural applications.

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

INTRODUCTION

Alor Regency is an archipelagic region with relatively expansive agricultural land that farmers can utilize for cultivation. Agricultural development in Alor has shown positive progress, but crop production there remains heavily dependent on fertilizers (Hermita Putri et al., 2019; Zakarias Mautuka, Astriana Maifa, 2022). The community primarily uses urea fertilizer to enrich their crops. Urea is widely employed because it accelerates plant growth, making it a preferred choice for agricultural land. One of the key elements in urea fertilizer is nitrogen (N). If plants lack nitrogen, they cannot grow normally; therefore, nitrogen is essential for plant growth and serves as a primary building block for plant proteins. On the other hand, urea is highly soluble in water and prone to volatilization into the air. Plant absorption of urea is not optimal, as only about 30–40 % of applied nitrogen is absorbed by plants, while the remaining 60 % is lost as ammonia gas through volatilization. Due to its high solubility in water, urea cannot be efficiently absorbed by plants (Kuczuk, 2019; Liu et al., 2023).

Urea is classified as a fertilizer containing 46% nitrogen (N). Nitrogen is a macronutrient that plants require in relatively large amounts and plays a vital role in greening and photosynthesis (Liu et al., 2023; Rosa et al., 2018). Micronutrients, though needed in much smaller quantities, are nonetheless essential for physiological processes and plant growth. These nutrients are considered essential. Essential micronutrients such as Fe, Mn, Zn, B, and Cu include iron (Fe), which plays a critical role in plant respiration and chlorophyll formation. If plants lack iron, they become infertile or withered. Iron can be sourced from iron sand of Puntaru that contains 77,80% Fe (Karbeka et al., 2021; Martasiana Karbeka, Faryda Veronica Lamma Koly, 2020). The addition of Fe to a urea solution can support plant growth. However, excessive Fe can be toxic to plants, causing direct cellular damage, destroying lipids and proteins, and impairing root growth thus necessitating controlled Fe release in the environment. One approach to enhance the absorption efficiency of both nitrogen from urea and Fe is to reduce solubility by using a matrix to coat the fertilizer. This coating process for urea and Fe can utilize environmentally friendly materials via the slow-release fertilizer (SRF) method, which controls or slows the release of nutrients for plant growth(Sholeha et al., 2024). One promising material for SRF production is chitosan. Chitosan is a natural polymer widely used as a microcapsule wall material because it is biodegradable and biocompatible. It has advantages such as good degradability, non-toxicity, environmental and living organism safety, and possesses antimicrobial, antifungal, and metal-chelating properties (Herianus et al., 2024). However, chitosan also has drawbacks, including low mechanical strength when forming hydrogels—resulting in low water absorption (swelling). Therefore, chitosan must be modified with other materials to improve its mechanical properties. One such material that can serve as a matrix to enhance chitosan's mechanical properties is biochar (Das et al., 2022; Lestari et al., 2022; Martasiana Karbeka, Zakarias Mautua, 2024).

Biochar refers to a carbon-rich solid produced from biomass through incomplete combustion under minimal oxygen conditions (pyrolysis). Biocar is not ably more stable in soil and resists oxidation. The advantages of using biochar include its role as a soil conditioner enhancing soil stability, improving permeability and aeration, boosting soil organic carbon content, and increasing the retention of nutrients and water for plant use. Biochar is utilized in slow-release fertilizers due to its long-term persistence in soil and its function as a method for storing soil carbon critical for maintaining soil fertility (Manzoor et al., 2022; Zakarias Mautuka, Astriana Maifa, 2022).

Implementing chitosan-biochar composites as coatings for urea and iron (Fe) involves experimenting with various compositions to achieve optimal properties for slow-release fertilizer applications (Yu et al., 2024). Determining the optimal bead composition includes testing water absorption capacity and analyzing phase composition and crystal structure using X-ray Diffraction (XRD).

MATERIALS AND METHODS

Materials

The instruments used in this study include a volumetric flask, graduated cylinder, Erlenmeyer flask, dropper pipette, analytical balance, glass beaker, magnetic stirrer, chemistry glassware, and stirring rod. Samples were analyzed using X-Ray Diffraction (XRD). The materials used in this research were iron sand, urea, biochar, chitosan, 6% acetic acid, distilled water (aquadest), NaOH, and a pH indicator.

Procedures

Preparation of the Chitosan Solution

Five grams of chitosan powder were transfered into an Erlenmeyer flask. Then, a 2% acetic acid solution was added, and homogenized the mixture using a magnetic stirrer until the chitosan dissolves completely, forming a hydrogel.

Synthesis of Slow-Release Fertilizer Beads

The slow-release fertilizer was prepared using several modified treatments. First, chitosan was mixed with 2% acetic acid and stirred with a magnetic stirrer for 30 minutes. The chitosan solution was then divided into four portions, and each was supplemented with urea, biochar, and Fe in the following variations:

Table 1. Composition Variations.

Sample code	Fe (g)	Urea (g)	Biochar (g)
CB_2U_2Fe	1	2	2
CB_3U_2Fe	1	2	3
CB_2U_3Fe	1	3	2
CB_3U_3Fe	1	3	3

Note: Sample code description
CB_2U_2Fe **: 2 g biochar - 2 g urea**
CB_3U_2Fe **: 3 g biochar - 2 g urea**
CB_2U_3Fe **: 2 g biochar - 3 g urea**
CB_3U_3Fe **: 3 g biochar - 3 g urea**

Before adding Fe, urea, and biochar into the chitosan solution, dissolve urea in 15 mL of water, then add the specified amount of biochar and stir using a magnetic stirrer until the mixture is homogeneous. Next, introduce the urea solution into the chitosan, then stir for 10–15 minutes until homogeneous. Add Fe and mix with a spatula. The resulting solution is ready to be molded by injecting it into a 1 M NaOH solution using a syringe. The resulting beads are basic, so they must be neutralized by washing with water until the pH is neutral. Confirmation that the beads are neutralized is done via negative tests with pH paper and indicators (PP), then dried.

Water Absorption Test

The water absorption test followed a modified procedure from reference (15). Each variant of the slow-release fertilizer beads (0.5 g) was weighed and immersed in four glass beakers containing 25 mL of distilled water. Beads were soaked for 24, 48, 72, and 96 hours. After each specified soaking time, the beads were separated accordingly. The percentage swelling (% swelling) was calculated using the equation:

$$\%\text{Swelling} = \frac{Wwet - Wdry}{Wdry} x100$$

Characterization by FTIR and XRD

The chitosan–biochar–urea composite beads were characterized using FTIR and X-ray Diffraction (XRD). Samples were placed on a prepared sample holder, and diffraction angles (2θ) were set using Cu Kα radiation with a wavelength of 1.54056 Å. The test was run and the diffractogram data of the sample were obtained.

RESULTS AND DISCUSSION

Synthesis beads of chitosan–biochar–urea–Fe

The synthesis of chitosan–biochar–urea–Fe slow-release fertilizer beads begins with preparing a 2% acetic acid (CH_3COOH) solution to dissolve the chitosan powder, as illustrated in Figure 1.

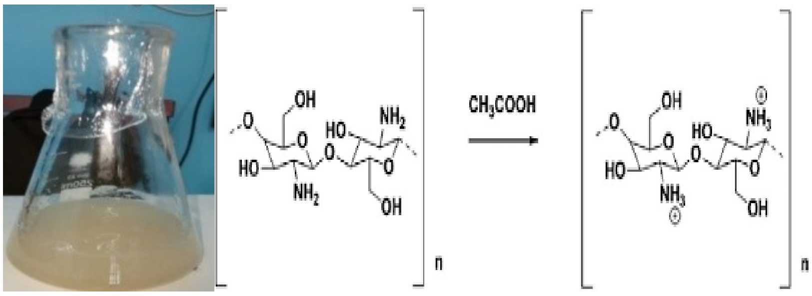


Figure 1. Reaction between Chitosan and Acetic Acid.

Chitosan is insoluble in water but dissolves well in acidic solvents, especially weak acids such as acetic acid. When chitosan is treated with acetic acid, a protonation reaction occurs where the $–NH_2$ groups are converted to $–NH_3^+$. This transforms the chitosan into a soluble form, resulting in a chitosan gel. Upon adding biochar, urea, and Fe to this chitosan gel and dripping the mixture into a NaOH solution, the gel solidifies, forming beads—illustrated in Figure 2.

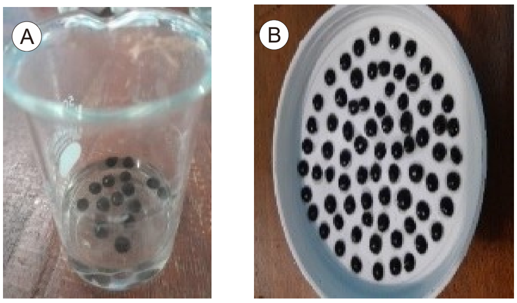


Figure 2. (a) Consolidation of chitosan–biochar–urea–Fe in NaOH. (b) The resulting beads exhibit a basic nature.

The compaction process occurs because chitosan undergoes deprotonation in an NaOH solution. Hydroxide ions (OH–) attack the amine groups on chitosan, causing the proton to be removed from the $–NH_3^+$ groups. This deprotonation increases inter-chain interactions among the polymer chains, resulting in a denser structure, as illustrated in Figure 3.

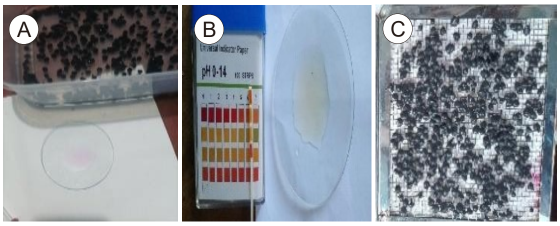
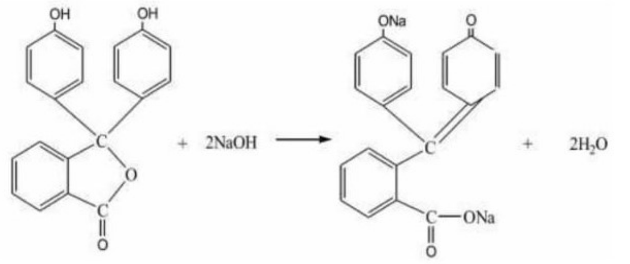


Figure 3. (a) Chitosan–biochar–urea–Fe beads in an alkaline state (basic), showing their initial formation; (b) Beads that have been neutralized; (c) Beads after washing to achieve neutral pH.

In addition, NaOH helps strengthen hydrogen bonds and ionic interactions within the gel network, contributing to its compaction. The resulting beads are basic in nature. Neutralization is achieved through repeated washing until neutrality is indicated by a color change in phenolphthalein (PP). In the first wash, the beads still show a basic character, evidenced by the appearance of red coloration. This red color in the filtrate occurs because residual NaOH reacts with phenolphthalein: the hydroxide ion (–OH) interacts with the carbonyl group on the phenolphthalein indicator, yielding a red color—illustrated in Figure 4.



PP indicator colorless (neutral) pink/red (alkaline).

Figure 4. Reaction of phenolphthalein indicator (PP) with NaOH:

Washing is continued until the beads reach neutrality, as indicated by a negative result with the phenolphthalein (PP) indicator—when PP is added, the filtrate remains clear with no color change. To further confirm that the beads are indeed neutral, the filtrate is also tested using universal pH indicator paper, which shows a neutral pH value (~pH 7).

Water Absorption (Swelling)

The water absorption test of the resulting beads aims to determine their ability to absorb water and assess their capacity as slow-release fertilizer beads made from chitosan, biochar, urea, and Fe. This test evaluates the material's ability to absorb water without undergoing fragmentation or lysis. Swelling occurs as water diffuses into the beads due to osmotic pressure and interacts with hydrophilic functional groups. Once equilibrium is reached, the absorbed water stabilizes. The results of the swelling test are presented in Figure 5.

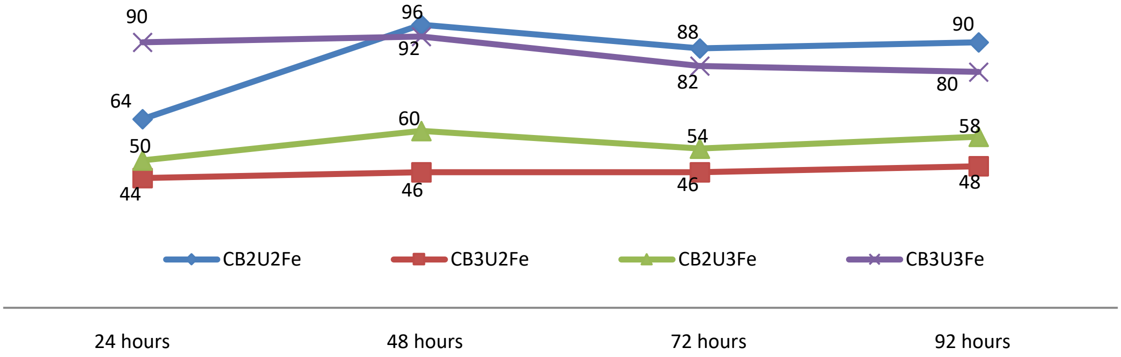


Figure 5. Percentage Water Absorption (Swelling).

Based on the swelling test results, among the four bead variants tested, beads labeled CB2U2Fe, CB2U3Fe, and CB3U3Fe reached an optimal swelling state at 48 hours. Swelling then declined at 72 hours before rising again at 92 hours, but this later increase did not surpass the 48-hour peak. In contrast, beads labeled CB3U2Fe continued to steadily increase their swelling percentage.

These findings indicate that during optimal swelling time, the beads likely reached saturation binding water through hydrogen bonding and occupying porous spaces—such that further exposure led to reduced absorption. Ultimately, the trapped water remained entrapped within the beads, causing them to swell further due to osmotic diffusion into hydrophilic groups and porous biochar entry.

Referring to Figure 5, the bead variants with relatively low water absorption CB3U2Fe and CB2U3Fe displayed maximum swelling percentages of approximately 48% and 60%, respectively. Meanwhile, bead variants CB2U2Fe and CB3U3Fe exhibited high water absorption capacity, reaching about 96% and 92%, respectively.

These differences in water absorption performance are influenced by the beads' composition. The variants CB3U2Fe and KB2U3Fe have biochar-to-urea ratios of 2:3, whereas CB2U2Fe and CB3U3Fe maintain a 1:1 ratio. The composition ratio affects cross-link formation via functional groups during bead fabrication: in CB3U2Fe and CB2U3Fe, the excess of one component fosters extensive cross-linking, resulting in tightly bonded polymer networks. This structure yields dense and strong bead matrices with enhanced hydrophobic characteristics which explains their lower water absorption. Conversely, beads with balanced ratios (CB2U2Fe and CB3U3Fe) develop fewer cross-links within the polymer matrix, resulting in a looser structure with more hydrophilic properties allowing significantly greater water uptake.

FTIR Characterization

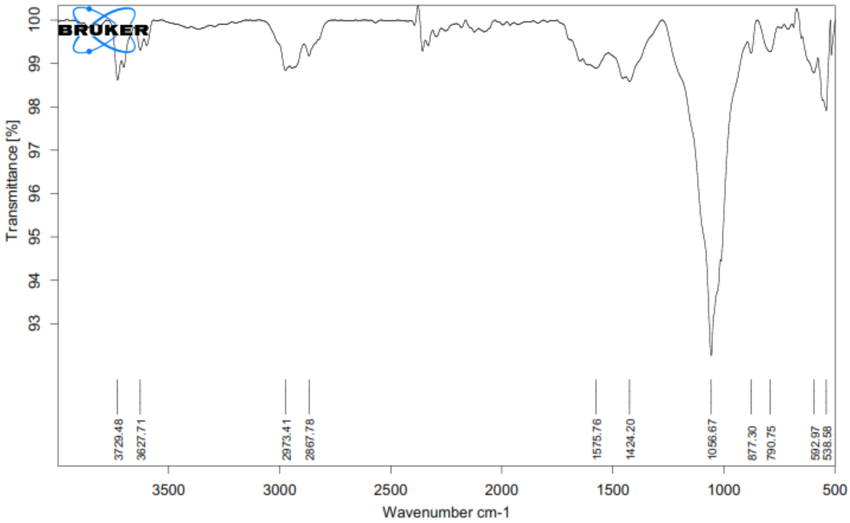


Table 2. Description of FTIR spectra.

No	Wavelenght (cm-1)	Functional group	Description
1	3729,48	Free O–H Stretching	The presence of free –OH groups in chitosan or biochar that have not fully reacted through hydrogen bonding formation.
2	3627,71		
3	2973,41	Stretching of C-H (aliphatic) or N-H group	Aliphatic C–H groups are found in biochar and chitosan, whereas N–H groups are commonly present in chitosan and urea
4	2867,78	Stretching C-H (aliphatic) typical organic compounds (methylene, CH2)	Characteristic organic functional groups are found in both chitosan and biochar.
5	1575,76	Stretching sy a zmmetrical -NH_2 group /N-H bending	The presence of vibrational modes of –NH_2/N–H groups—or N–H bonds—in urea or chitosan."
6	1424,20	Stretching C–N/ deformasi –CH_2	The presence of secondary amine groups and C–N vibrational modes are indicative of both urea and chitosan."
7	1056,67	Stretching C–O-C / C-O	Both functional groups originate from chitosan and biochar
8	877,30	Stretching C-H/Aromatic ring vibration	Can indicate the presence of conjugated aromatic structures
9	790,75	Weak Vibrations of Aromatic Rings	This vibration is likely originating from biochar
10	592,97	Metal Stretching –O (Fe–O)	Indicates the presence of iron oxide minerals in iron sand
11	538,58	Metal Stretching –O (Fe–O atau Si–O)	Characteristic Fe–O or possibly Si–O vibrations from biochar components

XRD Characterization

X-ray diffraction (XRD) analysis of the chitosan–biochar–urea–Fe composite fertilizer beads was conducted to determine their physical properties, including phase composition and crystalline structure. XRD was used to identify the crystallinity of the chitosan–biochar–urea–Fe composite. The results of this characterization can be seen in Figure 6.

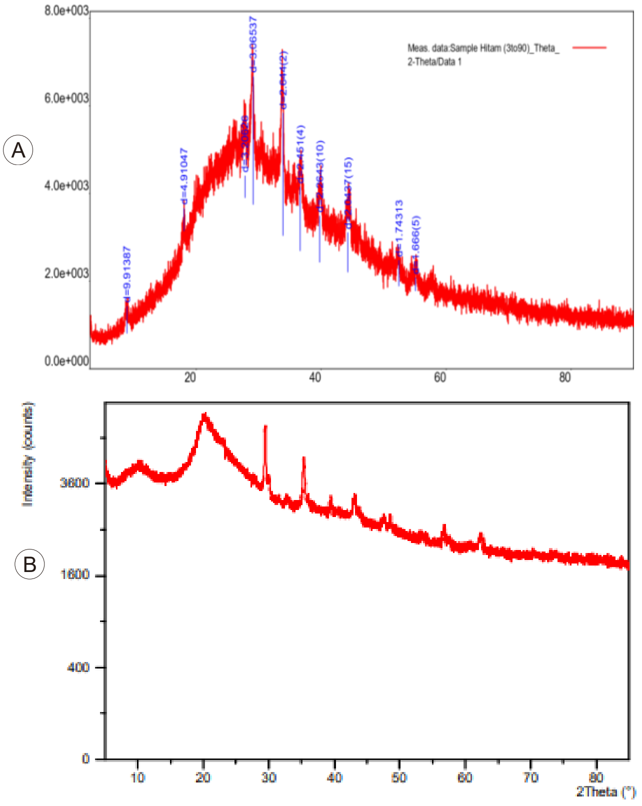


Figure 6. Diffractograms of the composite beads: (a) Chitosan–biochar–urea; (b) Chitosan–biochar–urea–FeDiscussion.

Based on Figure 6 (a), the diffractogram of the chitosan–biochar–urea composite beads indicates the presence of an amorphous phase, marked by a broad peak in the 2θ range of 20° to 60°. However, there are also distinct sharp peaks at 2θ = 29.1° and 33.9°, which correspond to the crystalline phase of urea. According to phase analysis from the diffractogram, the composite beads exhibit a semi-crystalline structure.

Based on Figure 6(b), the XRD diffractogram for the chitosan-biochar-urea-Fe composite beads shows a broad, amorphous halo around 2θ = 20°, indicating presence of a non-crystalline phase. However, there are also distinct sharp peaks in the 2θ range of 30°–40°, corresponding to crystalline phases of urea and Fe. Therefore, the chitosan-biochar-urea-Fe beads exhibit a semi-crystalline structure. Although both bead variants (with and without Fe) display semi-crystallinity, the diffractogram peaks suggest that the biochar-urea-Fe composition has a higher degree of crystallinity compared to the beads without Fe. Higher crystallinity is typically associated with enhanced mechanical strength, meaning that these beads would be more resistant to lysis or fragmentation, even when their water absorption (swelling) capacity is high. Thorough discussion represents the causal effect mainly explains for why and how the results of the research were taken place, and do not only re-express the mentioned results in the form of sentences, not repeat them. Concluding sentence should be given at the end of the discussion.

CONCLUSIONS

Based on the conducted study, the following conclusions can be drawn:

- Among the four synthesized variants of slow-release fertilizer beads, the ones with the highest water absorption capacity were CB2U2Fe, achieving 96%, and CB3U3Fe, with 92%.
- FTIR analysis confirmed the presence of CH, CN, NH, and Aromatic ring vibration groups characteristic of chitosan, biochar, and urea, along metal stretching -O groups associated with Fe, indicating successful bead synthesis.
- X-ray diffraction (XRD) analysis confirmed that the chitosan–biochar–urea–Fe composite beads exhibit a semi-crystalline structure.The conclusions of the study may be presented in here.

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

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