

Bio-based hydrogel fertilizer from h-collagen-g-PAA for water retention and urea management

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Abstract

Slow-release urea fertilizer (SRUF) with gradual nitrogen release and high water absorption capacity was synthesized in situ by incorporating urea into a superabsorbent hydrogel matrix of h-collagen-g-poly(acrylic acid). The water absorption capacity of the product was 110 (g/g) times its weight in distilled water at room temperature over 90 minutes. Nitrogen content analysis indicated that the product contained 5.58% nitrogen. The water-holding properties of the product and nitrogen-release behavior in soil and water media were also investigated. The findings indicate that the product exhibits good slow-release properties and excellent water retention capacity. This will efficiently enhance fertilizer utilization and water resource management simultaneously.

Keywords: *slow-release fertilizer, superabsorbent hydrogel polymer, urea, collagen hydrolysate.*

Data Availability: All data supporting the findings of this study are available from the corresponding author upon request.

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1. Introduction

The global agricultural sector faces challenges due to the excessive application of urea fertilizer and water scarcity during the dry season^[1,2]. Conventional urea fertilizer enhances soil nitrogen (N) content but is inefficient^[3,4]. Approximately 40-70% of N is lost to the environment or chemically bound in the soil, unavailable to plants, leading to economic losses, environmental pollution, and agroecological issues^[5-7]. Previous studies have shown that the use of slow-release fertilizers and superabsorbent hydrogel polymers can effectively address these issues^[8-11]. Integrating superabsorbent hydrogels (SAP) with fertilizers improves plant nutrition, minimizes the environmental impact of fertilizers, decreases evaporation, and reduces irrigation frequency^[12].

SAP are hydrophilic networks capable of absorbing and retaining significant quantities of water or aqueous solutions^[13,14]. Hydrogels function as reservoirs for water conservation that work near the root zone of plants. During irrigation, whether from irrigation water or rainwater, water can be collected, stored, and then released gradually to meet the plants' needs over an extended period after irrigation. Hydrogels combined with soil enhance air permeability, improve water absorption, and increase fertilizer retention, yielding economic advantages^[15]. However, using hydrogels in agriculture encounters multiple challenges, as most are composed of pure poly(sodium acrylate), which is costly,

non-biodegradable, and unsuitable for environments with water and soil salts^[16].

Multiple studies indicate that incorporating collagen hydrolysate (h-collagen) in the synthesis of superabsorbent hydrogel polymers enhances their swelling properties, lowers production costs, and results in biodegradable hydrogel polymers^[17-20]. H-collagen was selected as the hydrogel skeleton due to its biodegradability, abundance, and potential as a sustainable raw material derived from tannery waste. The present study is a continuation of our previous works on the extraction of collagen from leather trimming waste and the synthesis of superabsorbent hydrogels from the extracted collagen^[20,21]. Utilizing h-collagen derived from tanning industry by-products not only provides an environmentally friendly approach to waste management but also adds economic value to an otherwise discarded material, aligning with the principles of circular economy and sustainable agriculture.

Research on the coating of urea fertilizer with SAP polymers derived from h-collagen produced from leather industry solid waste has not been found in the literature. Bajpai and Giri^[22] stated that there are two methods for coating fertilizer with hydrogel. The first method entails incorporating the compound (fertilizer) into the reaction mixture, where polymerization occurs in situ, encapsulating the fertilizer within the gel matrix. The second method allows the dry polymer gel to swell in a fertilizer solution.

Once equilibrium is achieved, the gel is dried, producing a superabsorbent that contains fertilizer^[16]. This study employed the first method due to its greater efficiency in implementation.

Therefore, this study aimed to develop a biodegradable slow-release urea fertilizer (SRUF) synthesized in situ using h-collagen as a natural biopolymer backbone. The main objectives were to: (1) fabricate an h-collagen-based SRUF composite, (2) evaluate its swelling behavior, water retention, and nitrogen release characteristics, and (3) analyze its release kinetics using established kinetic models. The significance of this research lies in introducing a sustainable, waste-derived biopolymer for fertilizer encapsulation, which not only reduces nutrient loss and improves water management in soil but also promotes the valorization of tannery waste, contributing to circular bioeconomy and sustainable agricultural practices.

2. Materials and Methods

2.1 Tools and materials

The materials used were h-collagen, methylene bisacrylamide (MBA), potassium persulfate (KPS), acrylic acid (AA), 2-acrylamido-2-methylpropane sulfonic acid (AMPS), distilled water, ethanol, Merck urea (p.a), and ultisol soil. The soil was obtained from the research field of the Faculty of Agriculture, Lambung Mangkurat University, located at Jl. Unlam III, Banjarbaru City, South Kalimantan. The collected soil was cleaned of leaves and non-soil materials, sieved through a 26-mesh sieve and air-dried overnight. H-collagen was synthesized following the method described in our previous study^[21]. The equipment used included a three-neck reactor, a magnetic stirrer, a thermostatic water bath, a vacuum apparatus, scissors, an oven, nylon cloth, and polyester gauze.

2.2 Production of slow-release urea fertilizer (SRUF) urea in situ

This method was based on modifications of Purba et al.^[20]. H-collagen (1.33 g) was dissolved in 40 mL of distilled water, placed in a three-neck reactor equipped with a mechanical stirrer (300 rpm) and immersed in a thermostatic water bath at 80 °C. Subsequently, a certain amount of 70% neutralized AA (4.7 g in 5 mL H₂O) and 2 g of urea were added and stirred for 10 minutes. After that, a crosslinker solution (0.14 g MBA in 5 mL H₂O) and an initiator solution (0.15 g KPS in 5 mL H₂O) were added successively. The reaction was conducted for 60 minutes at 80 °C and 300 rpm. The entire reaction was conducted under vacuum conditions. After the reaction was complete, the gel formed was poured into excess ethanol (200 mL) and left to stand overnight. The product was then reduced in size using scissors, washed with 200 mL of fresh ethanol, and filtered. The product was dried in an oven at 50 °C for one night. After drying, the sample was stored away from moisture, heat, and light. Three independent batches of SRUF were synthesized to account for experimental variability.

2.3 Water absorption measurement

One gram of SRUF was immersed in tap water and left to soak at room temperature for 90 minutes. The swollen

SRUF was filtered through an 80-mesh sieve to eliminate unabsorbed water and subsequently weighed. Water absorption was calculated using Equation 1. Each measurement was performed in triplicate, and the average value was reported.

$$WA = \frac{M}{M_0} - 1 \quad (1)$$

where: M indicates the weight of swollen SRUF; M₀ indicates the weight of dry SRUF; and WA is the water absorption capacity per gram of dry SRUF.

2.4 Maximum water holding capacity of soil

SRUF sample (1 g) was mixed with 100 g of dry ultisol soil (under 26 mesh) and placed in a PVC tube with a diameter of 4.5 cm. The bottom of the tube was sealed with nylon cloth (with an aperture of 0.076 mm) and weighed (marked W1). The soil sample was slowly moistened with tap water from the top of the tube until water seeped out from the bottom. The tube was reweighed (marked W2) when no water seeped from the bottom. A control experiment was conducted without SRUF. The maximum water holding ratio (Wt %) of the soil was determined using Equation 2. The experiment was conducted with three replicates for each treatment (with and without SRUF).

$$wr\% = 100(W2 - W1) / (W2 - W1 + 200) \quad (2)$$

2.5 Urea release in soil media

Ten grams of filtered soil were mixed with 1 gram of SRUF. The mixture was placed in a 5 cm diameter PVC tube lined with nylon cloth at the bottom and fitted with a device to collect liquid. Regular watering with 20 mL of distilled water was conducted daily for 10 days. The solution that had passed through the soil (leachate) was collected daily to analyze the total nitrogen value using the micro-Kjeldahl method. Urea release in soil was tested in triplicate.

2.6 Urea release in water media

A total of 0.5 grams of SRUF was placed in a beaker glass filled with 1000 mL of distilled water. Water samples of 2 mL were collected at intervals of 0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 60, 90, and 120 minutes. The water samples were placed in glass containers and stored at 5 °C until the next nitrogen (total N) concentration test. Each test was repeated three times independently, and the average results were reported.

2.7 Nitrogen Release kinetics

Release kinetics can describe the rate of nitrogen release and its release model. This is an important parameter in SRUF because it determines its effectiveness. This study evaluated nitrogen release kinetics using four models: Zero Order, First Order, Higuchi Order, and the Korsmeyer-Peppas model. Kinetic parameters were calculated based on the mean values obtained from three replicate experiments.

2.8 Zero order reaction kinetics

In this zero-order system, nitrogen is released at a constant rate, regardless of concentration. The zero-order release

system is ideal for SRUF (sustained release). Zero-order kinetics follow Equation 3^[23]. The graph derived from this equation represents a linear function. In zero-order kinetics, the dissolution profile is slow due to the absence of system separation, assuming that the dissolution area remains under equilibrium conditions^[23]. Zero-order kinetics is obtained by plotting the cumulative percentage of nitrogen release against time^[24,25].

$$Q_1 = Q_0 + k_0t \quad (3)$$

where: Q_1 indicates nitrogen concentration; k_0 indicates zero-order constant; and t is time.

2.9 Release kinetics of the Korsmeyer-Peppas model

This kinetic model is founded on Fick's law. It describes the release of a solute when a combination mechanism occurs between Fickian diffusion transport and non-Fickian Case II transport controlled by polymer chain relaxation. This model is commonly applied to analyze the release of a substance in a polymeric system^[23]. Kottegoda et al.^[25] indicate that the graph produced by this method illustrates the relationship between time and the fractional release of nitrogen, plotted as the logarithm of nitrogen release against the logarithm of time. The kinetics of the Korsmeyer-Peppas model is described by Equation 4.

$$\frac{M}{M_\infty} = k \quad (4)$$

3. Results and Discussions

3.1 Preliminary research

The preliminary study evaluated two methods for the production of SRUF. The initial method employed by Sarkar et al.^[26] consisted of coating urea with dry hydrogel. This method necessitated the use of a rotary drum to ensure even distribution of the hydrogel powder on the surface of the urea granules. Due to the unavailability of the rotary drum in the laboratory, it was substituted with a simpler device that involved stirring in a closed container using a clockwise rotating motion. The resulting SRUF exhibited an irregular size. The second method evaluated was a modification of the approaches by Purba et al.^[20]. This method is efficient in terms of time, as urea is incorporated during the hydrogel formation process, which is why it is also known as in situ synthesis. This method yields SRUF with a more consistent size compared to the prior method. This method was chosen for the production of SRUF.

3.2 Nitrogen release in soil media

One of the most important characteristics of the SRUF is its slow-release property. The nitrogen release rate in the soil medium is presented as accumulation (Figure 1). The nitrogen content, calculated using the micro-Kjeldahl method, is expressed as a percentage (%) and converted into cumulative nitrogen (g). The cumulative value represents the quantity of nitrogen that can be released from the produced SRUF. In addition, measurements were also conducted on nitrogen release from pure urea fertilizer (Merck p.a).

The release of nitrogen from pure urea fertilizer is faster than that from SRUF. Nitrogen is highly volatile and easily washed away, potentially leading to environmental pollution that disrupts ecosystem balance^[27,28]. Experimental results showed that conventional urea released approximately 91% of its total nitrogen content within 10 days, whereas the SRUF released only about 47% over the same period. Unlike urea fertilizer, the SRUF generated in this study could release nitrogen slowly and consistently. The slower the nitrogen release, the smaller the fertilizer loss due to evaporation and leaching. Therefore, plants are expected to absorb the nitrogen contained in SRUF optimally. In addition to its slow nitrogen release capability, the SRUF exhibits a swelling ratio of 110.8 g/g when tested in distilled water.

The mechanism by which urea is slowly released from SRUF in the soil is as follows. SRUF will gradually expand as water is added to the soil, transforming into hydrogel. This causes an increase in the size of the three-dimensional layer openings and allows the diffusion of fertilizer solution in the hydrogel network. Urea in SRUF is slowly dissolved by water in the swollen hydrogel network. Additionally, urea release is influenced by dynamic exchange between free water in the hydrogel and soil water^[29-31].

According to recent studies the nitrogen release behavior of polymer-based slow-release fertilizers is mainly influenced by water diffusion, polymer relaxation, and matrix degradation^[32,33]. The hydrogel network formed by h-collagen, acrylic acid, and AMPS contains hydrophilic functional groups ($-\text{COOH}$, $-\text{NH}_2$, and $-\text{SO}_3\text{H}$) that establish hydrogen bonding and electrostatic interactions with urea molecules. These interactions temporarily immobilize urea within the network, delaying its diffusion. Meanwhile, the hydrogen-bonded water molecules within the hydrogel enhance the material's water retention and holding capacity, providing a moist microenvironment that facilitates gradual nutrient release. As the hydrogel undergoes repeated swelling and deswelling cycles, the network relaxation and partial degradation further promote a sustained and predictable nitrogen release profile, consistent with diffusion-controlled and polymer-relaxation mechanisms reported in the literature.

3.3 Kinetic model of nitrogen release in soil media

The kinetics of nitrogen release from SRUF were estimated by plotting a curve between the cumulative amount of nitrogen and time. These values were then compared with the kinetics of nitrogen release from pure urea.

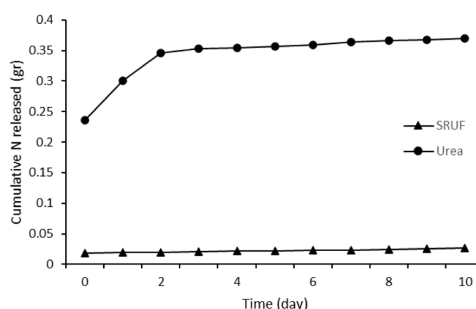


Figure 1. Graph of nitrogen release in soil medium.

The nitrogen release data were fitted to Zero-order, First-order, Higuchi, and Korsmeyer–Peppas models to determine the best-fit release kinetics based on linear regression analysis. The best-fitting model was determined based on the coefficient of determination (R^2) of each regression, which indicates the proportion of total variation in the dependent variable explained by the independent variable, with values ranging from 0 to 1^[34]. A model is deemed a good fit when R^2 approaches one. Figure 2 displays the kinetic graphs for each tested model.

The values of the release rate coefficient (k) and diffusion exponent (n) were obtained from the processing of dissolution data into the equation $y = a + bx$. For zero-order kinetics, b was the parameter; for first-order kinetics, it was $-b/2.303$; the Higuchi order was represented by b ; and in the Korsmeyer-Peppas model, n equaled $b^{[35]}$. Table 1 summarizes the equations and R^2 values for each kinetic model tested. The data in Table 1 indicate that the highest R^2 value for SRUF achieved in this study, approaching one, corresponds to the zero-order release kinetics model. Therefore, the Zero-Order model is highly suitable for describing the nitrogen release kinetics from SRUF. Nitrogen release following Zero-Order kinetics exhibits a nitrogen release rate independent of the initial nitrogen concentration in SRUF and remains constant over time^[36,37].

3.4 Water holding capacity of ultisol soil

The maximum water-holding ratio of Ultisol soil was significantly improved by the incorporation of SRUF. In soil without SRUF, the maximum water-holding ratio was 20.89%, whereas the addition of SRUF at a mass ratio of 1:100 (SRUF to soil) increased this value to 46.67%.

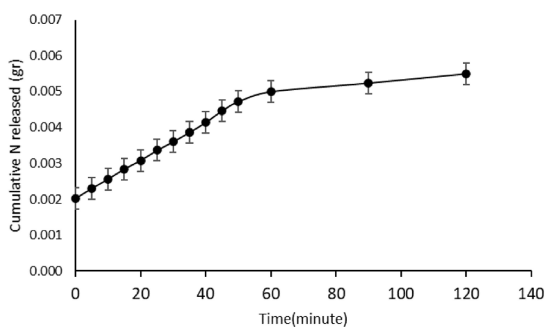


Figure 2. Graph of nitrogen release from SRUF in water.

This represents a 25.78% enhancement in water retention compared with the control soil. The improvement achieved in this study exceeds those reported in previous works. For instance, Guo et al.^[2] reported a 12.45% increase in water-holding capacity with polymer-coated fertilizers, and Guo et al.^[38] observed an 11.3% increase.

The SRUF produced in this study demonstrates a high water absorption capacity in the soil, enhances the soil’s water holding capacity, and effectively stores rainwater or irrigation water, thus improving the efficiency of water resource utilization. This value exceeds that reported in earlier studies, representing a notable advantage over traditional slow-release fertilizers.

Similar mechanisms have been reported in recent studies, where hydrogen bonding, polymer relaxation, and reversible swelling–deswelling cycles play a major role in sustaining water retention in polymer-based fertilizers^[32,33]. Moreover, the incorporation of h-collagen contributes to improved structural flexibility and mechanical stability, allowing repeated swelling and shrinking without disintegration. This synergistic behavior enhances both the durability and efficiency of the SRUF in regulating soil water dynamics.

Overall, the SRUF developed in this study demonstrates a substantial improvement in the soil’s ability to retain and regulate water compared with conventional fertilizers. This improvement supports sustainable soil–water management practices and offers a promising approach to improving fertilizer efficiency under varying environmental conditions.

3.5 Morphological observation of SRUF before and after water absorption and soil incubation

Morphological observations were conducted to visually support the functional performance of SRUF, particularly in terms of water absorption and nitrogen release mechanisms. Figure 3A shows the SRUF in its dry state, appearing as firm, opaque, and compact granules. After being immersed in distilled water for 90 minutes (Figure 3B), the SRUF exhibited a substantial increase in volume, forming a transparent hydrogel structure. This morphological transformation indicates the material’s strong hydrophilic nature and confirms the crosslinked hydrogel network’s ability to absorb and retain large quantities of water. Following ten days of incubation in Ultisol soil with daily watering (Figure 3C), the SRUF maintained its swollen hydrogel form but showed partial surface degradation and a darker coloration. These changes suggest water exchange between the hydrogel and

Table 1. Kinetic model of nitrogen release in soil media.

Type	Parameter	Kinetic Model of Nitrogen Release			
		Zero Order	First Order	Higuchi	Korsmeyer-Peppas
SRUF	Line equation	$y = 0.0008x + 0.0181$	$y = 0.0162x - 1.7374$	$y = 0.0027x + 0.0166$	$y = 0.1423x - 1.7409$
	R^2	0.9967	0.9932	0.9264	0.9222
	k	0.0008	-0.0070	0.0027	1.3877
	n	-	-	-	0.1423
Pure Urea	Line equation	$y = 0.0093x + 0.2963$	$y = 0.013x + 0.5332$	$y = 0.0386x + 0.2641$	$y = 0.0748x + 0.5011$
	R^2	0.5869	0.5706	0.8332	0.8395
	k	0.0093	-0.0056	0.0386	1.188
	n	-	-	-	0.0748

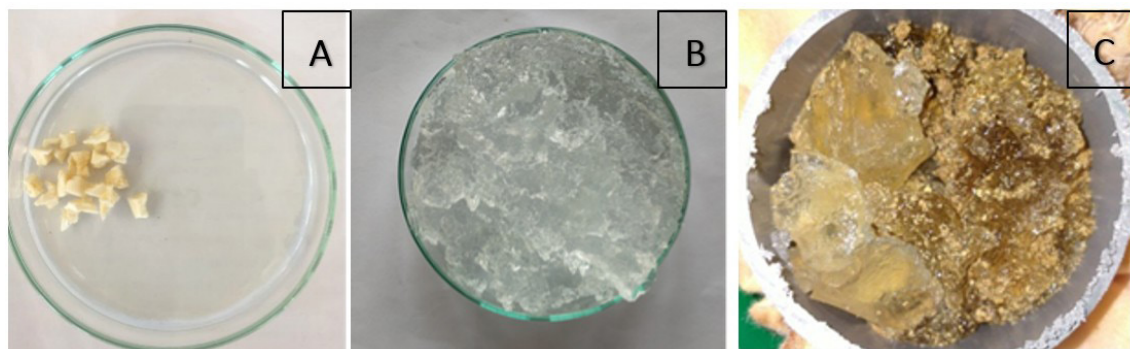


Figure 3. Morphological appearance of SRUF under different conditions: (A) dried SRUF before swelling; (B) SRUF after water absorption for 90 minutes; (C) SRUF after 10 days of soil incubation.

Table 2. Kinetic model of nitrogen release in water media.

Parameter	Kinetic Model of Nitrogen Release			
	Zero Order	First Order	Higuchi	Korsmeyer-Peppas
Line equation	$y = 0.00003x + 0.0026$	$y = 0.0036x - 2.5808$	$y = 0.0004x + 0.0017$	$y = 0.3133x - 2.8877$
R ²	0.8425	0.7632	0.9362	0.9559
k	0.00003	-0.00156	0.0004	2.0573
n	-	-	-	0.3133

the surrounding soil environment, consistent with nitrogen diffusion from the SRUF matrix to the soil solution. The partial degradation further demonstrates the biodegradable characteristics of the collagen-based hydrogel, which aligns with the sustainable design objective of this material.

These visual observations are consistent with previous findings on collagen-based hydrogels, where morphological changes during swelling and soil interaction are attributed to the diffusion-controlled release of urea and gradual polymer relaxation within the hydrogel matrix^[32,33].

3.6 Nitrogen release in water media

The nitrogen release profile of SRUF in distilled water at room temperature indicates that 20% of nitrogen was released into the medium within 120 minutes. As illustrated in Figure 2, SRUF exhibited a steady and gradual release pattern, indicating that the hydrogel matrix was able to withstand the internal pressure generated by the dissolution of urea and maintain its structural integrity during immersion. This behavior demonstrates that the nitrogen release in aqueous conditions is predominantly governed by diffusion through the swollen hydrogel network rather than by rapid dissolution of urea.

3.7 Kinetic model of nitrogen release in water media

The kinetic graphs of each tested model are presented in Figure 2. A summary of the equations, R² values, and n values of each kinetic model tested in water are presented in Table 2. The nitrogen release profile of SRUF in water tends to follow the Korsmeyer-Peppas kinetic release model with an R² value closest to 1. In the Korsmeyer-Peppas equation, the parameter n characterizes the mechanism of nitrogen release. Table 3 illustrates the relationship between n and the nitrogen release mechanism. Table 2 shows that

Table 3. Relationship between the release exponent (n) and the release mechanism.

Release exponent (n)	Release mechanism
< 0.5	Quasi-Fickian
0.5	Fickian
0.5 < n < 1	Anomalous (non-Fickian)
1	Non-Fickian case II
> 1	Non-Fickian super case-II

the value of n = 0.3133 indicating that the nitrogen release mechanism from SRUF in water is based on quasi-Fickian diffusion. This suggests that the release behavior is controlled by a combination of water penetration into the hydrogel and the diffusion of urea through the partially relaxed polymeric chains.

4. Conclusions

SRUF derived from the coating of urea with the superabsorbent hydrogel polymer h-collagen-g-poly(acrylic acid) has been successfully produced. SRUF contains 5.58% nitrogen with a water absorption capacity of approximately 110.8 (g/g) of its weight in distilled water. SRUF has a water retention capacity of 27.78% higher than water without SRUF. Nitrogen release from SRUF in soil medium is significantly slower compared to pure urea fertilizer. SRUF releases approximately 45% of nitrogen over a period of 10 days, whereas urea fertilizer releases 83%. Thus, the SRUF produced in this study can be classified as a slow-release fertilizer. In water medium testing, SRUF releases approximately 47% of nitrogen over 120 minutes. Based on the release kinetics observed, there are differences in kinetic models between SRUF produced and urea fertilizer.

The release kinetics of SRUF in soil medium follow the Zero-Order kinetic model with an R2 value of 0.9996. In contrast, it follows the Korsmeyer-Peppas model in a water medium with an R2 value of 0.9559.

5. Author's Contribution

- **Conceptualization** – Febriani Purba
- **Data curation** – Febriani Purba
- **Formal analysis** – Febriani Purba
- **Funding acquisition** – Ono Suparno
- **Investigation** – Febriani Purba
- **Methodology** – Febriani Purba
- **Project administration** – Febriani Purba
- **Resources** – Febriani Purba
- **Software** – NA.
- **Supervision** – Ono Suparno; Meika Syahbana Rusli; Is Fatimah
- **Validation** – Ono Suparno; Meika Syahbana Rusli; Is Fatimah
- **Visualization** – Febriani Purba
- **Writing – original draft** – Febriani Purba.
- **Writing – review & editing** – Febriani Purba; Ono Suparno; Meika Syahbana Rusli, Is Fatimah.

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