



Original Research Article

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## Modified Chitosan–Polyvinyl Alcohol Membrane as Environmentally Friendly Slow-Release Urea Fertilizer

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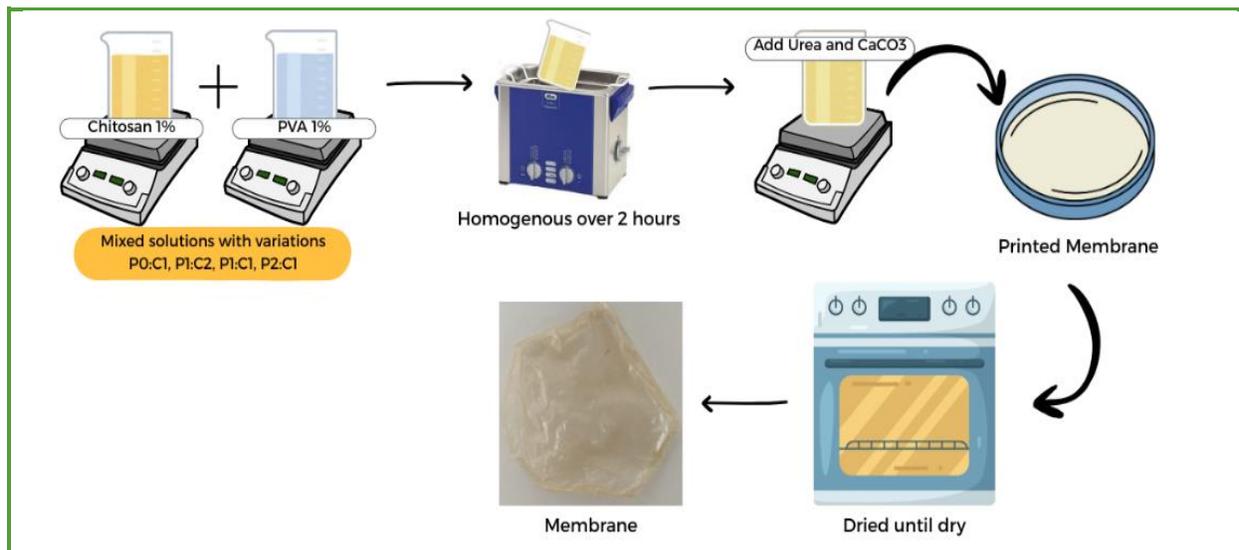
Controlled nutrient

### ABSTRACT

Slow-release fertilizers (SRFs) can improve nutrient use efficiency and reduce environmental impacts. In this study, an environmentally friendly SRF was prepared using chitosan, polyvinyl alcohol (PVA), urea, and calcium, aiming to enhance urea absorption by plants. The membrane was fabricated using the casting method, in which the polymer solution was poured into Petri dishes and the solvent was evaporated to obtain a dry SRF membrane. The results showed that increasing the PVA content in the SRF matrix reduced the porosity from  $9.3 \times 10^{-4}$  to  $5.4 \times 10^{-4}$  and increased the swelling degree from 105% to 121%. Scanning electron microscopy revealed pores on the membrane surface, allowing gradual urea release through diffusion. The release kinetics of samples P0, P1, and P2 followed the Korsmeyer–Peppas model, while P3 followed first-order kinetics. These findings indicate that the chitosan–PVA membrane can serve as an effective slow-release urea fertilizer, promoting plant growth while being environmentally sustainable.

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## Graphical Abstract



## Introduction

One of the inorganic fertilizers commonly used in agriculture is urea fertilizer with a nitrogen content of 46%, which plays a role in the formation and growth of plant parts, such as chlorophyll formation, and accelerates the growth of leaves and roots to be suboptimal [1]. However, urea fertilizer has low fertilizer use efficiency because the release of nitrogen into the soil occurs rapidly, causing the nitrogen content absorbed by plant roots to not be maximized [2]. Nitrogen released by urea in the form of nitrate can cause environmental pollution, as nitrate can easily seep underground and reach groundwater and surface water, rendering the groundwater unsuitable for consumption if the nitrate levels exceed the permitted limits [3]. Water and soil pollution caused by low urea efficiency can be overcome by physically coating urea fertilizer granules with organic/inorganic materials that have hydrophobic properties that function as walls or diffusion barriers [4], so that urea fertilizer can be maximally absorbed and can supply nutrients to plants slowly. Physically coating urea fertilizer is known as Slow-Release Fertilizer (SRF). SRF

can release the elements in the fertilizer gradually and can adjust to meet nutrient demands during plant growth. The use of the SRF method is designed to create a physical barrier to nutrient transport with materials that can prevent water from diffusing, thereby reducing urea fertilizer emissions and increasing nutrient utilization by plants [5]. SRF can be made from biopolymers combined with other composite materials, as biopolymers can serve as a barrier matrix to prolong fertilizer release time [6].

One biopolymer that is widely used in various fields is chitosan. Chitosan is versatile as a functional material that can form composites with other materials [7]. In addition, chitosan also has hydrophobic properties and can form films. These properties can serve as effective walls or diffusion barriers, but chitosan has weak mechanical properties, so it needs to be combined with other materials that will provide additional functional properties [8]. Based on research [9], chitosan combined with montmorillonite can increase its release percentage when compared to chitosan alone. However, montmorillonite, which is a clay mineral, can undergo compaction under certain

conditions, which can reduce its permeability. Therefore, it is necessary to combine chitosan with other materials. One material that can be used is polyvinyl alcohol (PVA), as PVA has superior hydrophilic properties due to the presence of  $-OH$  groups, allowing PVA to enhance water absorption [10]. According to research [11], as the concentration of PVA increases, the degree of swelling produced will also increase because the hydrophilic groups of PVA will form hydrogen bonds with other polymer groups, enabling water molecules to easily penetrate the pores. The combination of chitosan and PVA is most recommended for improving film-forming ability. When the two polymers are mixed, hydrogen bonds will form between their functional groups, namely  $-NH_2$  and  $-OH$  in both polymers, resulting in improved mechanical properties of the matrix. According to Vo *et al.* [12], the combination of chitosan and PVA with a cross-linking agent will produce SRF with a low urea release capacity. Therefore, the addition of a cross-linking agent is not recommended in the chitosan-PVA matrix. It is necessary to add other materials that serve to increase bond strength while still allowing for controlled fertilizer. This material is abundant, non-toxic, and has high stability, making it suitable for environmental applications. The material that can be used is  $CaCO_3$  [13]. According to Stanley *et al.* [14], the addition of Ca material in combination with PVA-alginate can reduce its hydrophilicity because Ca will agglomerate, thereby acting as a barrier to reduce the rate of water diffusion, matrix dissolution, and urea release. This occurs because  $CaCO_3$  ionizes into  $Ca^{2+}$  ions, which subsequently form complexes with chitosan due to the binding between chitosan and  $Ca^{2+}$  ions [15].

Based on the above background, it is necessary to produce SRF fertilizer to increase

the nitrogen absorption capacity of urea fertilizer.

## Experimental

In this study, several materials were used, such as distilled water, glacial acetic acid, 1% (w/v) chitosan, polyvinyl alcohol,  $CaCO_3$ , urea, para-dimethylaminobenzaldehyde (*p*-DAB), and NaOH.

### Methods

#### SRF manufacturing

Membrane-shaped fertilizer is made by mixing PVA solution with chitosan solution in ratios of 1:2, 1:1, and 2:1 (v/v) stirred using a magnetic stirrer at a temperature of 60 °C at a speed of 300 rpm for 24 h. The homogeneous mixture was then sonicated for 20 min at 60 °C. After that, 0.01 g of  $CaCO_3$  and 0.2 g of urea were added to the solution and stirred for 2 h until homogeneous. The homogeneous mixture was then sonicated for 15 min at 30 °C. Next, 10 mL of the resultant solution was taken, then poured into a Petri dish, and dried at 65 °C until dry.

The dried and formed membrane was then soaked in a 1 M NaOH solution until the membrane could be removed from the Petri dish. The membrane was then rinsed with distilled water until the pH was neutral [16]. The formula for each urea SRF membrane is in Table 1.

#### SRF characterization

##### Swelling test

The swelling test in this study was conducted by weighing the dry weight of the membrane using an analytical balance, which was then immersed in 10 mL of distilled water for 6 h at room temperature. The immersed membrane was then dried with tissue and weighed as wet

**Table 1.** Membrane formula

No.	Membrane formula	Composition			
		Chitosan 1%	PVA 1%	Urea (ppm)	CaCO <sub>3</sub> (g)
1	P0	100 mL	0 mL	2000	0.01
2	P1	66.7 mL	33.3 mL	2000	0.01
3	P2	50 mL	50 mL	2000	0.01
4	P3	33.3 mL	66.7 mL	2000	0.01

weight. The following is Equation 1 for the degree of swelling, where WA is the degree of swelling, W<sub>s</sub> is the wet weight, and W<sub>d</sub> is the dry weight [17]:

$$WA = \frac{W_s}{W_d} \times 100\% \quad (1)$$

#### Porosity test

Porosity testing is carried out by weighing the dry weight of the membrane, then soaking the membrane in 10 mL of water for 6 h. The soaked membrane is weighed as wet weight, then dried using an oven at 100 °C until the membrane is dry and weighed as dry weight after being in the oven. The porosity of the membrane can be calculated using Equation 2, where  $\varepsilon$  is the percentage porosity, W<sub>s</sub> is the wet weight, W<sub>d</sub> is the dry weight,  $\rho$  is the specific gravity of water, a is the surface area, and l is the thickness [18]:

$$\varepsilon = \frac{\omega_s - \omega_d}{\rho \times a \times l} \quad (2)$$

#### Surface morphology test

Surface morphology testing in this study used SEM instruments.

#### Functional group test

Functional group testing was performed using FTIR instruments.

#### Nitrogen release test

This was done by weighing membranes of equal weight, which were then immersed in 100 mL of aquadess. The results of the immersion were tested using a UV-Vis spectrophotometer to determine the urea content. The immersion was tested on days 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, 24, 26, 28, 30, 32, 34, 36, 38, and 40 using DMAB reagent [19]. The data will be analyzed using Equations 3-6, zero-order, first-order, Higuchi, and Korsmeyer-Peppas kinetic models:

$$\text{Zero Order } Q_t = K_0 t \quad (3)$$

$$\text{First Order } \log \frac{Q_t}{Q_0} = \frac{K_1 t}{2.303} \quad (4)$$

$$\text{Korsmeyer-Peppas } Q_t = K_{kp} t^n \quad (5)$$

$$\text{Higuchi } Q_t = K_H t^{1/2} \quad (6)$$

#### Plant testing

This was done by measuring the weight of the membrane before it was placed in the soil, then preparing the planting medium with the same amount in each pot. The plants planted must be of the same height and number of leaves. Planting was carried out with the roots of the plants touching the membrane. After that, the plants were observed for leaf length, number of leaves, and stem length. These observations were carried out once a day. Data collection on the plants was observed for 14 days [20].

## Results and Discussion

SRF urea synthesis was carried out to obtain SRF urea in membrane form. The stages of SRF urea fertilizer synthesis began with the dissolution of chitosan using acetic acid and distilled water to dissolve PVA. The resulting chitosan and PVA solution was then mixed to produce an SRF membrane. The resulting mixture of chitosan and PVA solutions were then mixed with  $\text{CaCO}_3$  and urea to form SRF membranes with varying volume ratios between PVA and chitosan, resulting in variations of PVA 0 (P0), PVA 1:2 (P1), PVA 1:1 (P2), and PVA 2:1 (P3).

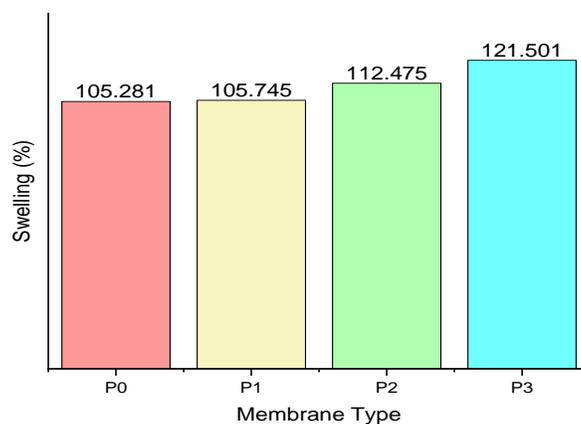
From [Figure 1](#), it can be seen that the SRF urea fertilizer produced has differences in its membrane layers. Fertilizers with variations P0 and P1 produce fertilizers that have thicker layers than P2 and P3. It can be noted that P0 and P1 are less transparent compared to P2 and P3, which have clearer membrane layers. The thin membrane layer of the P3 variation makes the membrane difficult to separate from the Petri dish, necessitating a longer immersion time in NaOH. Despite having a thin layer, the resulting membrane is not easily broken or torn. The SRF urea fertilizer produced is relatively elastic and not easily damaged.



**Figure 1.** Membrane type

### Swelling analysis

The water absorption (swelling) characteristics of the membrane were characterized to determine the water absorption strength of each SRF membrane variation. The measurement results showed the degree of membrane swelling for each variation presented in [Figure 2](#). The results of the swelling degree indicate that as the concentration of PVA increases, the swelling degree also increases. The results show that variation P3 has the highest swelling degree, namely 121.501%. Swelling or water absorption testing demonstrates that the more PVA contained in the SRF membrane composition, the higher the degree of swelling.



**Figure 2.** Swelling test diagram

This is because PVA has hydrophilic properties from its -OH groups that are able to hold water molecules through hydrogen interactions [21]. This makes the membrane capable of absorbing water from the surrounding environment.

#### Porosity analysis

Membrane thickness characterization was performed to determine the membrane thickness for each SRF variation. The measurement results showed the membrane thickness for each variation presented in Figure 3. Based on the porosity percentage data, the variation with the smallest porosity percentage is P3. This variation has the smallest PVA concentration among variations P1 and P2. However, variation P0, or without PVA, has the largest porosity of 0.00093%. The porosity percentage results show that the higher the PVA composition, the smaller the porosity percentage of the membrane. This may be due to the addition of PVA in the modified chitosan SRF

matrix, filling the pores in the membrane, thereby reducing the size and number of pores and decreasing. PVA can fill the pores in the membrane because the hydrophilic nature of PVA gives it the ability to gel, forming a gel network when it dries, making the SRF more compact, and PVA can fill the pores in the membrane. The hydrogen bond interaction between urea, chitosan, and PVA produces a denser surface structure with low porosity [12]. The decrease in porosity will affect inhibiting urea release, so even though this SRF membrane has a fairly high degree of swelling, urea release will remain controlled due to its decreased porosity value.

#### Surface morphology analysis

Surface morphology was characterized using SEM to visually determine the surface morphology of the membrane (Figure 4). The SEM results supported the porosity data because pores formed in the membrane could be observed on the membrane surface.

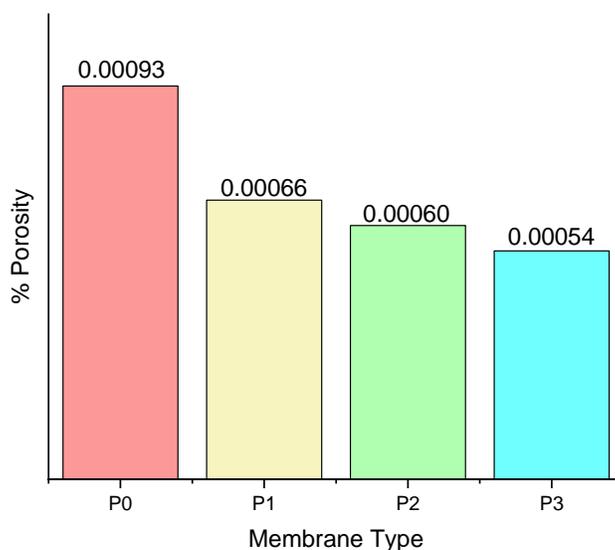
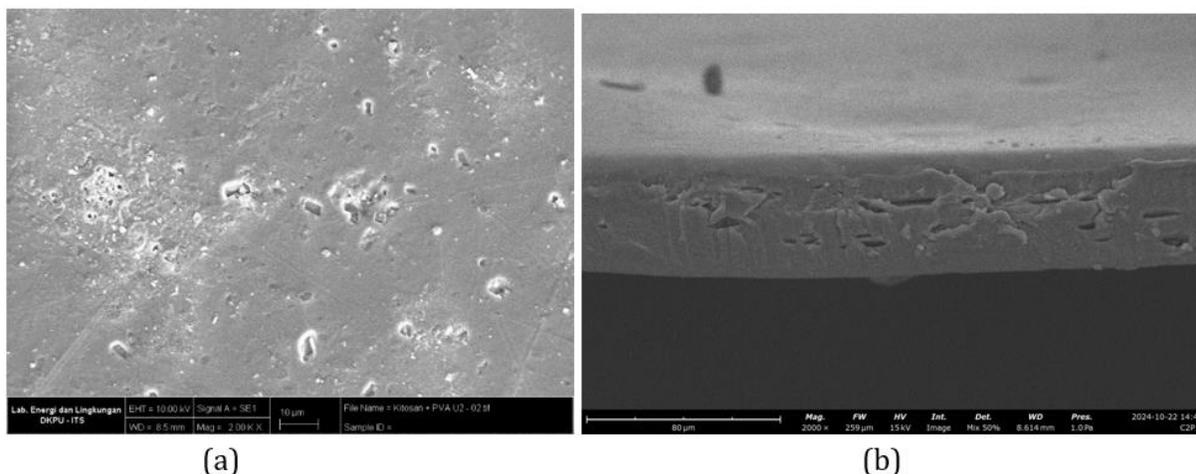


Figure 3. Porosity test diagram



**Figure 4.** Membrane morphology (a) surface and (b) cross-section

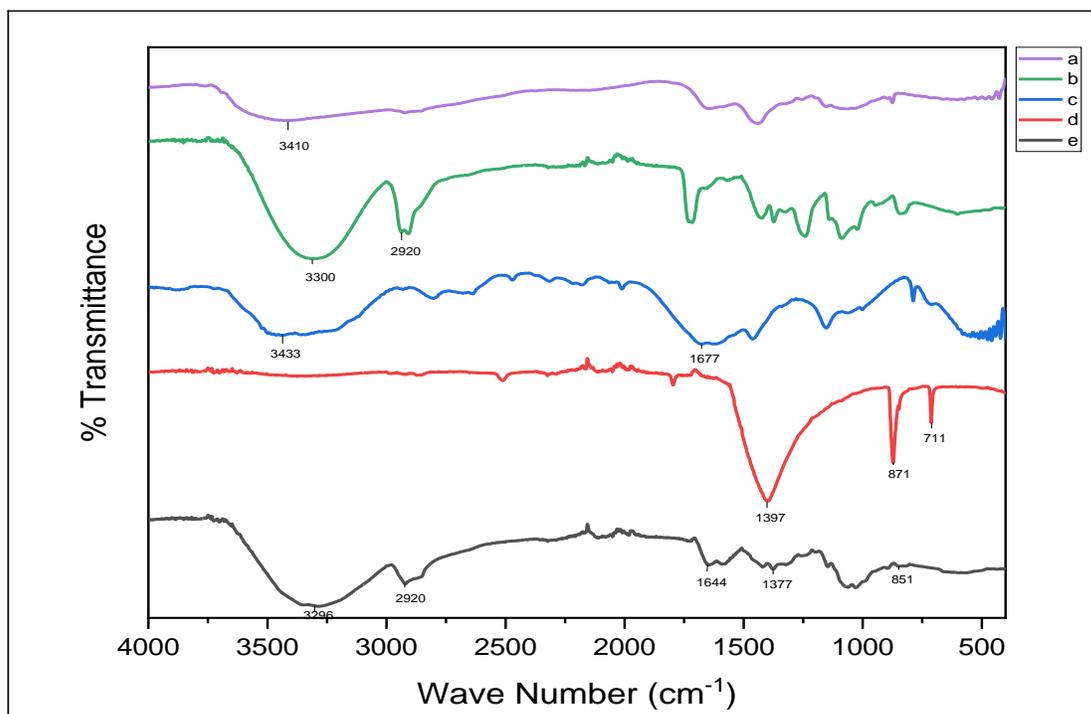
This test was conducted at a magnification of 2,000 times. The SEM test results showed that the surface morphology of the membrane exhibited a fairly diverse pore shape with an uneven membrane surface. Thus, it can be concluded that the synthesized SRF urea fertilizer can form pores and can be used as a slow-release urea fertilizer.

#### *Functional group analysis*

Functional group testing aims to identify changes in chemical structure that occur as a result of the synthesis process. The resulting IR spectra will be presented in the form of an overlay of chitosan, PVA, urea,  $\text{CaCO}_3$ , and SRF membrane to identify the functional group characteristics of each material and the changes that occur during the synthesis process. The IR overlay results are shown in [Figure 5](#).

[Figure 5](#) shows the IR overlay results of chitosan, PVA, urea,  $\text{CaCO}_3$ , and SRF membranes. From [Figure 5](#), the functional groups that appear in the SRF membrane can be identified, namely -OH, N-H, C-H, C-OH, and C=O groups. [Table 2](#) presents the wave number data obtained from FTIR analysis of the SRF membrane. Based on

the results of functional group identification of the SRF membrane, there was a shift in the absorption bands of the -OH, N-H, C-OH, and C=O groups. The shift in the absorption band at  $3,296 \text{ cm}^{-1}$ , which indicates the presence of strain vibrations from the hydroxyl (-OH) and amine (N-H) groups, shows the presence of hydrogen interactions between the H atom of chitosan and the O atom of PVA [22]. The absorption band of native chitosan is at  $3,410 \text{ cm}^{-1}$ , and that of PVA is at  $3,300 \text{ cm}^{-1}$ . This indicates a shift in the absorption bands of the -OH and N-H groups resulting from the overlap between chitosan and PVA, producing a broader peak. Furthermore, an absorption band at  $2,920 \text{ cm}^{-1}$  was found, indicating the presence of C-H groups, which suggests the presence of hydrocarbon chains from the polymer matrix, consistent with the absorption band of PVA without any shift. The absorption band at  $1,644 \text{ cm}^{-1}$  indicates the presence of C=O group vibrations originating from the urea functional group. The absorption bands indicating the presence of  $\text{CaCO}_3$  are  $1,377 \text{ cm}^{-1}$  and  $851 \text{ cm}^{-1}$ . These absorption bands indicate the presence of vibrations from the C-OH group in the coordinate mode and C=O possessed by  $\text{CaCO}_3$  [23].



**Figure 5.** Overlay FTIR spectra((a) Chitosan, (b) PVA, (c) Urea, (d) CaCO<sub>3</sub>, and (e) SRF)

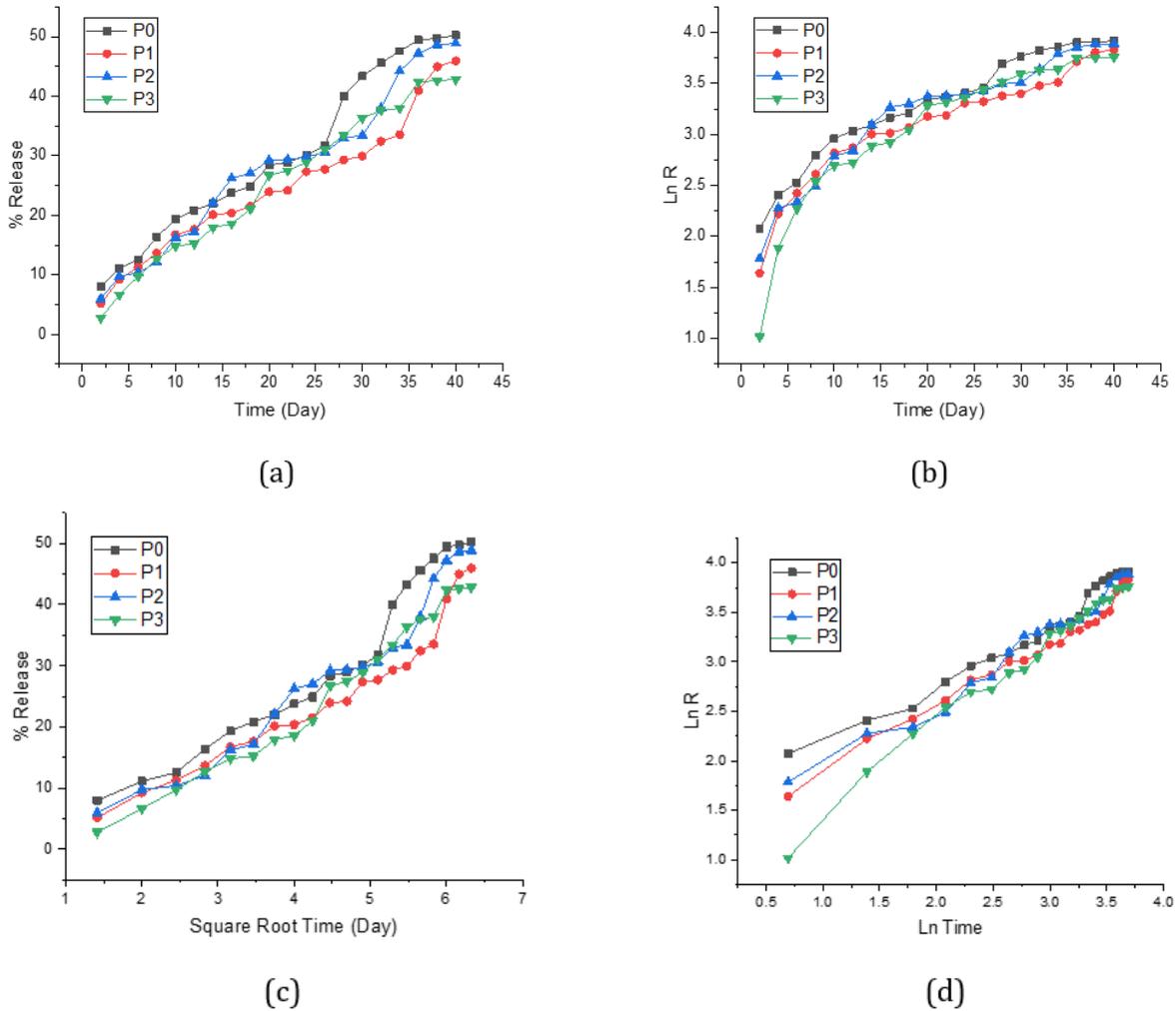
**Table 2.** Functional group

No.	Functional group	Wave number (cm <sup>-1</sup> )
1.	-OH, dan N-H	3296
2.	C-H	2920
3.	C-OH	1377
4.	C=O	1644
5.	C=O	851

### Nitrogen release analysis

Data analysis of nitrogen release from SRF on various kinetic equations is shown in [Figure 6](#). From the R<sup>2</sup> values obtained, the results show that the highest regression values are found in the Korsmeyer-Peppas release mechanism in variations P0, P1, and P2 with R<sup>2</sup> values of 0.978, 0.978, and 0.996, respectively. Therefore, it can be said that the urea release mechanism follows Korsmeyer-Peppas with non-Fickian diffusion because it has a value of  $0.45 < n < 0.89$ , indicating

that urea release does not follow the normal diffusion law due to obstacles in the diffusion process [24]. In the P3 variation, urea release follows a first-order mechanism, which can be interpreted as urea release in P3. The longer the urea is released, the less it is released in accordance with the remaining urea concentration. This occurs because P3 has low porosity, making it more difficult for water to enter the SRF, resulting in a reduced rate of dissolution and diffusion of the released urea ([Table 3](#)).



**Figure 6.** Kinetic models ((a) Zero order, (b) First order, (c) Korsmeyer-Peppas, and (d) Higuchi)

**Table 3.** Kinetics fitting parameter of each mathematical model for urea release of membranes

Membrane type	Parameter	Kinetic model			
		Zero order	First order	Higuchi	Korsmeyer-Peppas
P0	R <sup>2</sup>	0.9681	0.9379	0.9432	0.9787
	k	5.7687	2.3601	-10.522	4.279
	n	-	-	-	0.6519
P1	R <sup>2</sup>	0.9725	0.8978	0.95	0.978
	k	4.7521	2.1945	-11.02	3.2056
	n	-	-	-	0.7199
P2	R <sup>2</sup>	0.9632	0.8893	0.9299	0.9961
	k	7.0175	2.1691	-8.0012	3.3471
	n	-	-	-	0.6692
P3	R <sup>2</sup>	0.9891	0.993	0.9792	0.8936
	k	2.9907	2.0741	-12.432	1.819
	n	-	-	-	0.872

### Plant testing analysis

Plant testing was conducted over a 14-day growing period (Figures 7-9). Measurements were taken of stem length and number of leaves for each variety, with overall length measured on day 14. The soil used in this test weighed 250 g per pot. The seeds purchased from Superindo were of good quality. From the results of testing the plants for 14 days, data on stem length (cm) were obtained. From the stem diagram, it can be seen that the plant with the longest stem is

variety P3. This can be attributed to the fact that it released the least amount of urea, so that the nitrogen in the urea released was more controlled, resulting in lush plants. According to the report [25], modified chitosan membranes with Ca can increase plant growth by enhancing the release capacity of the membrane. A statistical test was also conducted to determine whether there were significant differences between the varieties. The result was a sig. value  $<0.05$ , which means that there were significant differences in stem length between each variety.

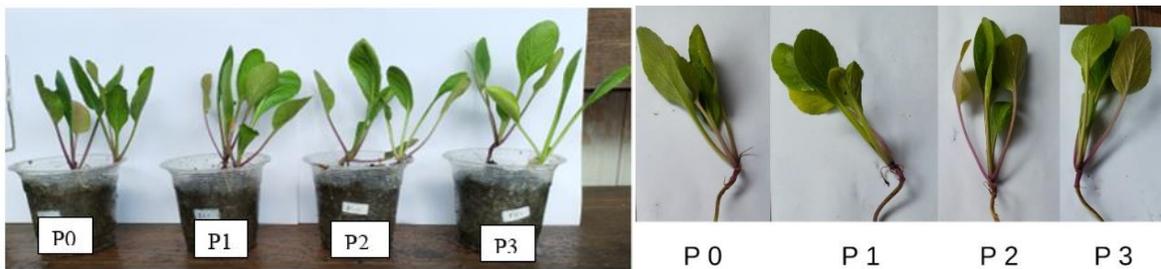


Figure 7. Plants testing

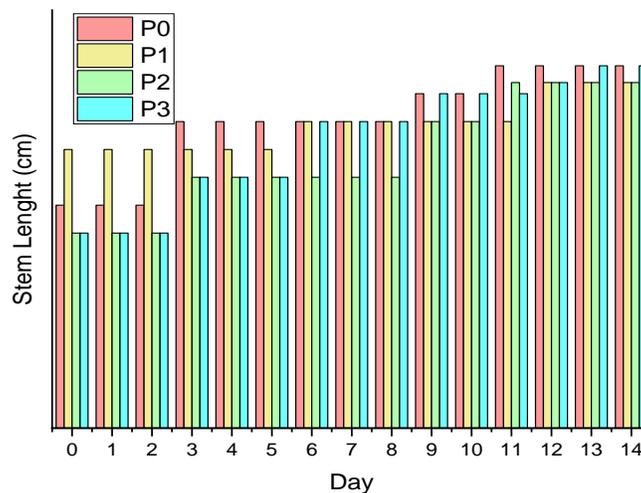


Figure 8. Stem length diagram

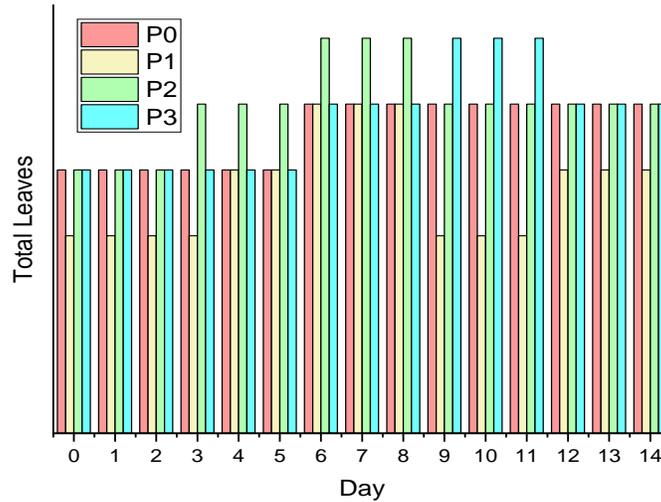


Figure 9. Total leaves diagram

## Conclusion

Based on the 14-day observation, all plant samples developed a relatively similar number of leaves, with P2 and P3 showing slightly higher leaf counts than the others. Statistical analysis revealed a significant difference in stem length among the treatments (sig. <0.05), indicating that the variations in fertilizer composition had a measurable effect on plant growth. This suggests that the chitosan-PVA-based slow release urea fertilizer can influence vegetative growth, with certain formulations promoting better development.

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## Conflict of Interest

The authors state that there is no conflict of interest to disclose.

## Authors' Contributions

All authors contributed to data analysis, drafting, and revising of the manuscript and agreed to be responsible for all aspects of this work.

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