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THE KINETIC STUDY OF CHITOSAN AND POLYVINYL ALCOHOL MODIFIED MEMBRANE FOR NPK SLOW-RELEASE FERTILIZER WITH CALCIUM CARBONATE

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ABSTRACT

A chitosan/poly(vinyl alcohol) (PVA) based slow-release fertiliser membrane system was developed to control sustainable nutrient delivery in a more environmentally friendly manner. The membranes were synthesised via blending with varying PVA compositions to modulate the polymer network structure and transport characteristics. Physical and morphological characterisations were conducted using mass and thickness measurements, swelling and porosity tests, as well as FTIR and SEM analysis. Nutrient release analysis focused on Nitrogen, quantified using the Ehrlich reagent method via UV-Vis spectrophotometry at a wavelength of 403 nm. The results indicated that increasing PVA concentration led to increased membrane porosity and swelling degree. The release kinetics were best described by the Korsmeyer-Peppas model, suggesting a mechanism governed by both diffusion and polymer relaxation. The C1:P1 composition exhibited the highest model fit with a coefficient of determination (R^2) of 0.9809, followed by C1:P2 (0.9646) and C1:P3 (0.8579). This study suggests that the lower PVA ratio (C1:P1) offers the most predictable kinetic control for sustainable agricultural applications.

Keywords: Chitosan, PVA, Membrane, NPK, Slow-Release Fertiliser.

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INTRODUCTION

Sustainable increases in food production are demanded by global population growth. The use of inorganic fertilisers is considered a key factor in this increase. Based on data from the 196th Edition of the DPR RI State Budget Bulletin in 2023, inorganic fertiliser was reported as the most widely used type, reaching 9.5 million tons in 2022. Inorganic fertilisers are widely utilised because they are more practical, easily available, affordable, and produce faster results due to the rapid release of nutrients, which helps plants grow more easily. One example of an inorganic fertiliser used is NPK(Nitrogen, Phosphorus, Potassium).
NPK fertiliser is widely recognised as the most used compound fertiliser. Global fertiliser usage is anticipated that it will hit 203.7 metric tons in 2024, according to a report by FAO. NPK fertiliser is utilized because nitrogen, potassium, and phosphorus, which are vital for the growth of plants, are contained within it.[1] Plant growth and photosynthesis are supported by nitrogen as a basic material, while energy transfer, root development, and plant reproduction are driven by phosphorus.[2] Furthermore, water relations, enzyme activity, and resistance to stress in plants are regulated by potasssium.[27,29] These elements are considered essential for plant growth. These elements are considered crucial for plant development. Instant nutrient availability, which results in rapid plant growth, and relatively low short-term costs are included among the advantages of NPK fertiliser. However, the use of conventional NPK fertilisers is deemed somewhat inefficient because the rate of nutrient release is observed to be quick. It is indicated by research that only 30% to 50% of fertiliser-derived nitrogen is typically utilised by

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vegetation. The remaining portion is dissipated into the environment via leaching, runoff, and atmospheric volatilisation, by which groundwater contamination and the emission of greenhouse gases are exacerbated.[16] Consequently, low nutrient use efficiency (NUE) is caused, while agricultural production costs and environmental impacts are increased.[4] The emergence of a modern agricultural model has been triggered by this problem, whereby the development of innovative slow-release fertilisers using a coating technology, such as Slow-Release Fertiliser (SRF), is encouraged to improve fertiliser efficiency. SRF is fabricated to slowly release nutrients gradually over time so that the required nutrients can be obtained by plants.[14] A semipermeable membrane is utilised by the SRF mechanism for coating,[23] and fertiliser components are integrated inside a matrix.[8] A barrier is established through molecular interactions as the primary idea of both methods, thereby preventing the facile escape of nutrients from the fertiliser granules into the environment.[13] However, synthetic polymer-based coatings, which are not environmentally friendly, are still relied upon for most commercial SRFs. This was demonstrated in the research by Isakov et al. (2025),[10,] where the application of synthetic polymers as coating materials for mineral fertiliser granules was examined. Although the rate of nutrient release is effectively controlled by these materials, high stability and the potential to leave environmental residues after long-term use are exhibited by synthetic polymers. In addition, the use of non-membrane granular coatings (sulfur-coated fertiliser) was explained in research by Witt et al. (2024)[28,] which were reported to be prone to cracking due to mechanical stress during storage, whereby uncontrolled nutrient release could potentially be caused. The development of ecologically acceptable fertiliser membranes that could efficiently regulate nutrient release was required to replace the usage of synthetic coated polymers in SRF, which are not biodegradable. In this research, the polymers used in the fertiliser membranes were chitosan and polyvinyl alcohol (PVA). Chitosan was used because of its biodegradable properties with amine and hydroxyl functional groups for molecular interaction with ions in nutrient control.[5] while PVA was used as a supporting polymer to form a chitosan-PVA composite membrane. The hydrophilic properties of PVA could increase the structural stability and mechanical resistance of chitosan membranes in controlled release systems.[23] In addition, calcium carbonate ($CaCO_3$) was added as an inorganic filler to improve the pore structure and increase thermal stability in regulating nutrient diffusion pathways, as well as to increase the supply of calcium, which is crucial for plant growth and development.[24] Thus, the development of NPK SRF fertiliser based on chitosan/PVA membranes with the addition of $CaCO_3$ was expected to produce a more efficient, biodegradable, and sustainable nutrient release system, as well as a reduction in the environmental impact of synthetic polymer leftovers.

EXPERIMENTAL

Materials and Instrumentations

Chitosan (MW = 132.812 g/mol; DD = >80%) derived from dried shrimp shells served as the primary membrane matrix in this experiment. PVA (30,000 g/mol), NPK fertiliser, NaOH (MW = 40 g/mol), calcium carbonate, glacial acetic acid (CH_3COOH; MW = 60 g/mol), HCl 37%, p-DMAB (MW = 149.19 g/mol), and distilled water were purchased from Merck. These investigations employed a Spectrophotometer UV-Vis Shimadzu 1700 (Japan) for nitrogen release testing, a Fourier Transform Infra-Red (FTIR) for the purpose of functional group analysis, and SEM for morphological analysis, which were tested at the Sepuluh Nopember Institute of Technology, Surabaya City.

Preparation of Chitosan 1% and 1000 ppm NPK Solution

One gram of chitosan was homogeneously dispersed in 100 mL of 1% aqueous CH_3COOH, followed by continuous magnetic stirring for a duration of two hours. To create the NPK stock solution (1000 ppm), 100 mL of distilled water and 0.1 g of fertiliser were combined, and the resulting mixture was uniformly stirred for an hour without the use of heat.

Synthesis of Chi/PVA/NPK

A 1% chitosan solution was combined with a 1% PVA solution in multiple volume proportions of 1:1, 1:2, and 1:3. These preparations were stirred at 400 rpm for ±24 hours at 55–60°C to achieve a homogeneous state. Following homogenization, the Chi/PVA solution was sonicated for ±20 minutes at 60 °C using the sweep method. The Chi/PVA solution was then split into two sections. Part A contained the Chi/PVA

solution, while part B contained the Chi/PVA solution with the addition of 1,000 ppm NPK solution and was stirred at a constant speed without heating.

Fabrication of Chi/PVA/NPK (SRF) Membrane

Three millilitres of the chitosan/PVA solution were deposited into a 5-cm Petri dish to establish the primary layer. Once prepared, the core layer was introduced by pouring 4 mL of the chitosan/PVA/NPK solution. For the last layer, a Petri dish was filled with three millilitres of the chitosan/PVA solution. The membrane was dried by oven-drying each layer at 60°C. A 1 M sodium hydroxide solution is used to soak the dry membrane until it separates. After that, distilled water is used to wash the membrane until the pH is steady. The membrane is then dried once again for 24 hours after being steeped in a $CaCO_3$ solution (0.01 g in 100 mL of distilled water and agitated without heat) for an hour at room temperature.

Membrane Characterization

Physical tests were used for the characterisation test. An analytical balance was used to weigh the membrane mass, and a Mitutoyo Thickness Meter was used to measure the membrane thickness. SEM was used for membrane morphological investigation, and FTIR was used for functional group identification in chemical tests.

Swelling and Porosity

After drying at room temperature, the membrane is cut into a 1x1 cm piece and weighed (W0). The membrane was then moistened for six hours at 25 °C in 10 mL of distilled water. After gently blotting the water-swollen membrane to remove any extra water surface area, the membrane is weighed using an analytical scale (W1). The swelling value is calculated using the formula below:

$$\text{Swelling } (\%) = \frac{W0}{W1} X100\%$$

A 1x1 cm membrane specimen was soaked in 10 mL of distilled water for six hours, dried, and weighed (W0) to determine its porosity. The membrane was placed in a drying oven set at 100°C for 24 hours. After cooling, the membrane was weighed to establish its ultimate weight (W1). Porosity is computed in this manner.

$$\text{Porosity } (\%) = \frac{W1 - W0}{\rho \times \alpha \times 1} X100\%$$

The density of distilled water (0.998 g/cm^2) is represented by ρ in the equation, the membrane's surface area (cm^2) by α, and the area (cm) by 1.

Slow-Release Test

For thirty days, 100 mL of water was used to soak each type of chitosan/PVA/NPK membrane. 5 mL of the soaking water was then removed every 2 days. Ehrlich's reagent (0.2 mL) was complexed with the soaking water in a vial bottle. The absorbance at 403 nanometers was then measured utilising a spectrophotometer UV-Vis. The released nitrogen content was calculated using the regression equation of the NPK standard solution. It was then examined using a release kinetic model (Korsmeyer-Peppas, Higuchi, first-order, and zero-order).

RESULTS AND DISCUSSION

Membrane Mass and Thickness

Based on Table-1, the mass and thickness of the membrane decreased with increasing PVA in the Chitosan-PVA ratio. The data indicated that PVA produced a lighter and thinner membrane by lowering the overall mass per unit area. The PVA had a higher molecular weight (30,000 g/mol) compared to chitosan monomers. Its high solubility enhanced polymer chain mobility, resulting in a more compact and less dense film. PVA produces a membrane that was lighter and thinner than chitosan by lowering the overall mass per unit area.[2] In contrast, positively charged chitosan exhibited a wider hydration layer that produced a thicker membrane structure. Therefore, the addition of PVA effectively suppressed excessive hydration of chitosan and promoted the formation of thinner membrane structures that were in line with previous research, indicating the role of PVA in improving membrane structural properties.[30]

Table-1: Membrane Weight and Thickness

Concentration	Weight(g)	Thickness ($x10^{-2}$ mm)
C1:P1	0.0726	0.0650
C1:P2	0.0693	0.0350
C1:P3	0.0612	0.0150

Functional Group Characterisation

FTIR spectra of NPK, $CaCO_3$, PVA, chitosan, and blank samples in the range of 4000 to 500 cm^{-1} are illustrated in Fig.-2. The NPK spectra in the 3517 cm^{-1} areas were interpreted as O-H stretching vibrations. The P-O and N-H stretching vibrations were confirmed by the areas at 3205 and 1440 cm^{-1}, respectively. In the meantime, the HO-P-OH bending vibrations were shown by the regions at 1086 and 540 cm^{-1}.[19] A spectral signal observed at 2846 cm^{-1} indicated a C-H alkane.[20] The presence of CH sp^3 and C-O stretching was demonstrated by the $CaCO_3$ spectrum's at 2517 and 1799 cm^{-1}, respectively.[5] The presence of C-O anti-symmetry stretch groups was demonstrated at 1042 cm^{-1}. In the meanwhile, calcite features were seen at 874 and 711 cm^{-1}.[9] The PVA spectrum at 3309 cm^{-1} represented OH stretching vibrations.[7] Absorption at 2941 and 2908 cm^{-1} was defined as C-H stretching. C=O stretching was indicated at 1725 cm^{-1}.[22] CH_2 bending and CH symmetric stretching were present in the areas at 1430 and 1372 cm^{-1}. Concurrently, C-O stretch vibrations were confirmed by an absorption band in the 1054 cm^{-1} range, and C-O stretching and C-C strain were confirmed by areas at 1090 and 834 cm^{-1}. The characteristic peak at the 876 cm^{-1} region was interpreted as C-N strain.[26] The spectral results of the samples were compared with the raw materials to observe the interactions between the components. The O-H group stretching vibrations from PVA, which overlapped with the N-H stretching vibrations from the primary amine groups of chitosan, were evidenced by a broad absorption band at 3296 cm^{-1}. A spectral signal observed at 2891 cm^{-1} was attributed to C-H stretching vibrations within the chitosan and PVA chains. The characteristic C=O stretching mode was observed as a prominent peak at 1648 cm^{-1}. [12] The characteristic peaks located at 1582 and 1416 cm^{-1} were interpreted as C-O and N-H bending.[18] Meanwhile, A spectral signal observed at 1153 and 1072 cm^{-1} corresponds to C-O stretching.[6]

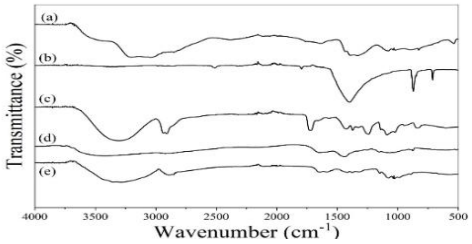
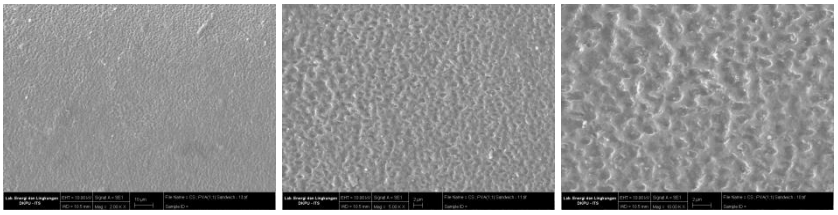


Fig.-1: FTIR Spectra of: (a) NPK, (b) $CaCO_3$, (c) PVA, (d) Chitosan, and (e) membrane sample

Morphological Characterization

SEM results showed that chitosan: PVA (1:1) composite exhibited a relatively homogeneous and microporous surface morphology. At 2,000x magnification (Fig.-2a), the membrane surface appeared continuous without visible macrocracks, while at 5,000x (Fig.-2b) and 10,000x (Fig.-2c), irregularly shaped micropores were evenly distributed, showing the formation of a consistent porous structure.



(a) (b) (c)

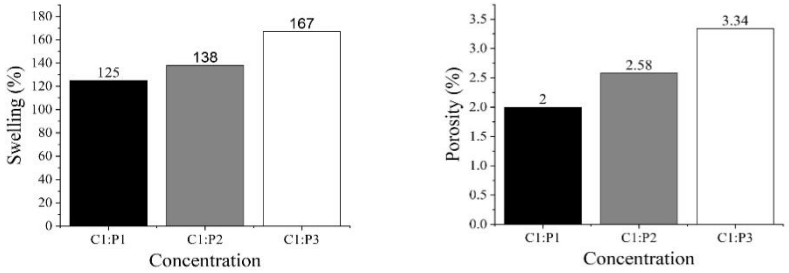
Fig.-2: (a) SEM C1:P1 Mag 2.00 K× (b) SEM C1:P1 Mag 5.00 K× (c) SEM C1:P1 Mag 10.00 K×

The formation of pores was attributed to H-bond interaction linking the -OH groups in PVA and the NH_2 or OH groups in chitosan, resulting in a less densely packed polymer network and the formation of micro gaps upon drying.[3] In addition, differences in structural characteristics, where PVA was more crystalline

and chitosan was more amorphous, contributed to incomplete chain compaction, further enhancing the formation of pores.[21] This porous morphology could improve membrane permeability and mass transfer performance.

Analysis of Membrane Swelling Mechanisms

Based on the Fig.-4a, the percentage of membrane swelling increased with increasing PVA concentration in the chitosan matrix. The C1:P3 concentration showed the highest %swelling, indicating enhanced hydrophilicity of the membrane. This behaviour was attributed to the dominance of -OH groups in PVA, which effectively bound water molecules. This was similar to previous research by Cahyaningrum *et al.* (2024)[2] which reported that membranes with higher PVA content exhibited greater swelling due to improved water diffusion and increased availability of hydrophilic interaction sites. Furthermore, adequate pore spacing allowed water to penetrate more easily into the membrane matrix, resulting in higher swelling values.



(a) (b)

Fig.-3: (a) Membrane Swelling Capacity, and (b) Membrane Porosity versus Membrane Concentration

Characterization of Membrane Porosity

It was evident from Fig.-4b that the percentage of membrane porosity increased with increasing PVA concentration in the chitosan matrix. The membrane with a C1:P1 ratio showed a porosity of ±2.00%, which increased to ±2.58% at C1:P2 and reached the highest value of ±3.34% at C1:P3. This was similar to research by Lusiana *et al.* (2024),[15,] which indicated that the addition of PVA contributes to the formation of a more porous membrane structure. This increase in porosity was attributed to the hydrophilic nature of PVA, which has -OH groups that can interact with the $-NH_2$ groups of chitosan through hydrogen bonding. This interaction reduced the distance between polymer chains and resulted in the formation of gaps inside the membrane matrix. As a result, higher PVA concentration enhanced pore formation, thereby increasing the membrane porosity and improving the permeability of the membrane.

Kinetic Release Profiles

Based Table-2, the coefficient of determination (R^2) for urea release decreased with increasing PVA concentration in the chitosan matrix, indicating a reduced fit to the Korsmeyer-Peppas model. The membrane with a C1:P1 ratio showed the highest R^2 value (0.9809), followed by C1:P2 (0.9646) and the lowest value at C1:P3 (0.8579). According to studies by Lusiana *et al.* (2024)[15] and Cahyaningrum et al. (2024),[2] adding high concentrations of PVA could reduce the effectiveness of nutrient release control.

Table-2: Kinetic Diffusion of NPK in Water Medium.

Models	Correlation coefficient (R^2)		
	C1:P1	C1:P2	C1:P3
Zero Order	0.9472	0.9463	0.9486
First Order	0.7617	0.7609	0.6246
Higuchi	0.7991	0.8075	0.8338
Korsmeyer-Peppas	0.9809	0.9646	0.8579

PVA with a C1:P1 ratio showed the highest R^2 of 0.9809, indicating the best release mechanism, namely the hydrogen bond interaction between the -OH (PVA) and $-NH_2$ (Chitosan) groups reaching an optimum point with the formation of cross-linking density, resulting in a tortuous diffusion path within the membrane matrix that inhibited nutrient diffusion through the membrane, making the nutrient release rate

slower and more stable. Meanwhile, PVA with higher concentrations (C1:P2 and C1:P3) showed lower R^2 values (0.9646 and 0.8579) as an increase in matrix hydrophilicity triggered a greater degree of swelling when in an aqueous medium.

CONCLUSION

This study successfully developed a chitosan/poly(vinyl alcohol) (PVA) based slow-release fertiliser membrane with tunable nutrient release characteristics through variation in PVA composition. An increase in PVA concentration has a significant effect on the membrane structure, as reflected by higher swelling and porosity levels, which reveal improved hydrophilicity and structural modification. FTIR and SEM analyses validated the existence of intermolecular interactions and morphological changes occurring within the membrane's internal matrix. The release kinetics were found to match the Korsmeyer–Peppas model, showing a diffusion-driven process affected by polymer chain relaxation. The C1:P1 compositions showed the optimal model fit with the highest coefficient of determination ($R^2 = 0.9809$) and more stable nutrient release behaviour. Therefore, the chitosan/PVA/$CaCO_3$ membranes with a lower PVA content (C1:P1) exhibit superior and more consistent performance in regulating nutrient release, making them a promising option for sustainable slow-release fertiliser (SRF) systems. This supports improved nutrient efficiency in agricultural practices, contributing to sustainable agriculture (SDG 2) and promoting responsible consumption and production (SDG 12) by minimising the waste of chemical resources and the use of bio-based materials.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTION

The present work is related to *SDG-12:Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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