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Formulation of Slow Release NPK Fertilizer Based on Cassava Starch and Lime Peel Biocomposite for Phosphate and Nitrate Leaching Reduction

Wanda Salsabila Maryam[1], Nina Hamidah[2]
[1]Chemistry Education Department, [2]Chemistry Department, Faculty of Tarbiyah and Teacher Training, UIN Sunan Kalijaga, Jl. Marsda Adisucipto No 1 Yogyakarta 55281, Indonesia. Tel. +62-274-540971, Fax. +62-274-519739.

Corresponding author*
wandaoux19@gmail.com

Abstract: Excessive nutrient loss from fertilizers is one of the main causes of water pollution and eutrophication in agricultural areas. To address this issue, the development of environmentally friendly Slow Release Fertilizers (SRF) has become increasingly important. Coating techniques can be applied to control the rate of nutrient release from fertilizers. The use of biodegradable coating materials is a top priority to reduce nutrient leaching that can potentially trigger water eutrophication. This study aims to formulate Slow Release Fertilizer (SRF) from NPK fertilizers with a coating matrix based on cassava starch and citric acid from lime peel, and to evaluate the rate of nutrient release behavior. Granular NPK-SRF was prepared by varying the mass composition of cassava starch and lime peel. The nutrient leaching rate was determined using UV-Vis spectrophotometry by measuring the nitrate and phosphate concentration in the leachate. The results showed that the coating mass composition greatly influenced the performance of SRF. The optimal formulation was obtained from a ratio of 78% cassava starch and 22% lime peel, which effectively reduced nitrate and phosphate leaching. This best formulation produced leachate with a NO_3- content of only 1.85 ppm and a PO_4^{3-} content of 0.56 ppm, which was much lower than other variations and the non-coated fertilizers. The findings demonstrate that the optimized cassava starch-lime peel coating matrix is an effective and prospective biodegradable coating materials for controlling phosphate and nitrate leaching and thus reducing the environmental risk of fertilizer application.

Keywords: Cassava starch; Eutrophication; Lime peel; NPK Fertilizer; Slow Release Fertilizer (SRF).

Introduction

Increased agricultural productivity in Indonesia is highly dependent on the use of inorganic fertilizers, particularly NPK fertilizers containing the macro nutrients nitrogen (N), phosphorus (P), and potassium (K). Although effective in supporting plant growth, excessive and uncontrolled use of inorganic fertilizers has had a negative impact on the environment, especially on water quality and the balance of aquatic ecosystems. One of the most significant impacts is the leaching of nitrate (NO_3^-) and phosphate (PO_4^{3-}) into water bodies, which leads to eutrophication (Akinnawo, 2023). This process is characterized by excessive growth of algae and aquatic plants, a decrease in dissolved oxygen levels, and disruption of the food chain in the water (Zulapriansyah et al., 2022). Data from the Ministry of Environment and Forestry (KLHK) shows that more than half of Indonesia's rivers are polluted, mostly due to agricultural fertilizer runoff (BPS, 2023). This condition emphasizes that conventional fertilization practices that rely on inorganic fertilizers need to be evaluated and replaced with more efficient and environmentally friendly systems. Therefore, the development of fertilizer technology that can release nutrients in a controlled manner is urgently needed

One innovation that addresses this challenge is slow release fertilizer (SRF). SRF has the ability to release nutrients gradually according to plant needs, thereby reducing nutrient loss due to

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leaching and increasing plant absorption efficiency (Savana et al., 2018).The effectiveness of SRF is highly dependent on the coating material used to control the rate of nutrient release. The use of natural, biodegradable, and non-toxic materials is key to ensuring that SRF formulations are in line with the principles of sustainable agriculture and green chemistry. Cassava starch is one of the natural materials with potential to be used as an SRF coating matrix. Indonesia, as one of the fourth largest cassava producers in the world, has abundant and sustainable starch availability (Ardyani et al., 2022).Cassava starch has biodegradable and biocompatible characteristics and is able to retain nutrient release for a longer period of time. However, its stability and water absorption capacity still need to be improved through a chemical modification (cross-linking) process using organic acids such as citric acid (Chamorro et al., 2024).

Natural sources of citric acid can be obtained from lime peel waste (Citrus aurantifolia), which contains high levels of citric acid, around 8.7% (Adindaputri et al., 2013)The combination of cassava starch and lime peel has the potential to produce a stable, biodegradable fertilizer coating biocomposite that is effective in reducing the rate of nitrate and phosphate leaching. The use of lime peel waste also provides added value in terms of horticultural waste management, thereby supporting the principles of circular economy and organic waste reduction in the environment.

Materials And Methods

Material

The materials used in this study included Natural Homemade Tapioca cassava starch, Pak Tani brand 16:16:16 NPK fertilizer, yard soil, lime peel (Citrus aurantiifolia), Cleo brand demineralized water, and cayenne pepper plants (Capsicum frutescens). Supporting materials used included black oil-based paint, Avian brand, and thinner.

Instrument

The instruments used in this study consisted of plant pots made from used gallons, a drill, a knife, an ISKU brand plant sprayer, and a brush. The measuring instruments used included a 250 mL measuring cup from One Two Cups, a Taffware brand food thermometer, scales, and a glass stirrer. The analytical instrument used in this study was a Thermo Scientific brand UV-Vis spectrophotometer.

Procedures

Lime Peel Preparation

The lime peel is first washed thoroughly and then drained. After that, the lime peel is dried at 50°C for 5 days at the Halal Innovation and Research Laboratory of Ahmad Dahlan University. The dried lime peel is then blended into a fine powder. The resulting lime peel powder is stored in an airtight container to keep it dry and ready for use in the next stage.

SRF Preparation

Cassava Starch Preparation (Gelatinization)

A total of 12, 10, and 14 grams of cassava starch were weighed as variations in material mass. Each starch mass was then gelatinized by adding ±50 mL of demineralized water. The starch and water mixture was heated while stirring constantly at a temperature of 70–80°C for ±10 minutes until a clear gel with high viscosity was formed, indicating that starch gelatinization had occurred optimally. After the heating process was complete, the starch gel was cooled to room temperature before being used in the next stage of SRF formulation.

Preparation of Planting Media

Soil samples were obtained from yards in Jambidan Kidul RT 05, Banguntapan, Bantul, Yogyakarta. Soil was collected at a depth of ±20 cm using a shovel. The collected soil was then weighed at 4 kg for each treatment. A total of four treatments were prepared according to the number of samples used in the study.

Mixing of Materials

NPK 16:16:16 fertilizer is weighed according to the mass variation of each sample. Dried and ground lime peel powder is then weighed based on mass variation. After the cassava starch gelatinization process reaches a stable gel condition, NPK fertilizer is added to the gelatin solution, followed by the addition of lime peel powder. The mixture is homogenized until it reaches a thick consistency, then cooled to room temperature. After thickening and hardening, the SRF material is molded, dried, and cut into small, uniform pieces before being applied to the growing medium.

Table 1 Variations in Slow Release Fertilizer Composition.

Growing Medium Code	Soil mass (kg)	NPK Fertilizer Mass (g)	Cassava Starch Mass (g)	Lime Peel Mass (g)
A	4	10	0	0
B	4	10	12	6
C	4	10	10	3
D	4	10	14	4

SRF Application

SRF media preparation

Four 15-liter used gallons were cut in the middle, then the upper part was used as a container. The container was given drainage holes spaced ±3 cm in a circle. Next, the surface of the gallon was coated with black matte oil paint that had been diluted with thinner, then aired until dry. Once dry, the top of the gallon was turned upside down, then a porous cloth with holes was attached to the bottom as a filter layer. Topsoil without fertilizer or compost was added on top of the cloth as the initial planting medium. Each gallon is given a different code, namely codes A, B, C, and D.

Application of SRF Fertilizer on Chili Plants

Cayenne pepper plants purchased at the Bumi Kencana Mandiri store, which were approximately 20 days old, were placed in gallons, then fertilizer that had been varied as shown in Table 1 was applied to each gallon. Fertilizer was applied once after 3 days of planting, and the plants were watered every morning and evening using demineralized water.

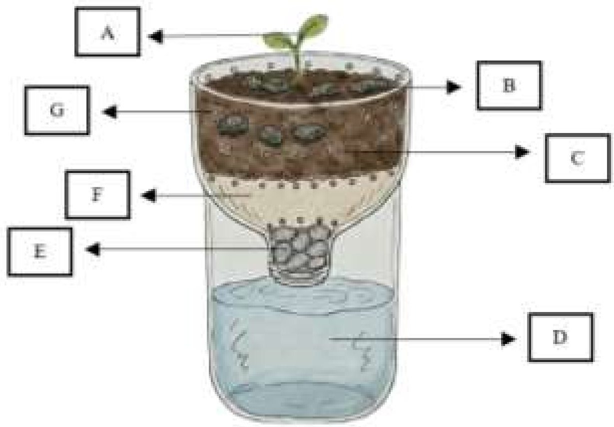

Figure 1 Application of SRF in growing media

Description:

Code A : Chili Pepper Plant
Code B : SRF
Code C : Topsoil
Code D :Fertilizer Runoff Water
Code E : Stones
Code F :Porous Fabric
Code G :Drainage holes

2.4 Analysis of Nitrate and Phosphate Levels

After 19 days, fertilizer runoff water or leachate from each gallon was taken as a sample to analyze the N-NO3- and N-PO43- levels at the Yogyakarta Research Instrument Standardization Agency (BSIP) Laboratory using a UV-Vis Spectrophotometer.

Results And Discussion

Physical Characteristics of Raw Materials (Lime Peel Powder) and Slow Release Fertilizer (SRF) Products

The process of drying sliced lime peel at a temperature of 50 °Cfor 5 days resulted in physical changes to the material. In terms of texture, the lime peel slices became dry and harder. Meanwhile, the color of the lime peel did not change significantly after drying. After the grinding and filtering process, a light green powder with a fine texture was obtained. This powder is then used as the main raw material for the formulation of Slow Release Fertilizer (SRF).

Meanwhile, Slow Release Fertilizer (SRF) formulations with varying concentrations produced clear differences in physical characteristics for each treatment code. Code A (Control) is the basic treatment without the addition of SRF. Meanwhile, Code B (High Concentration SRF) shows the most ideal physical characteristics, namely the densest texture, non-sticky, and easy to mold, as well as having a uniform deep green color. In contrast to Code B, Code C (Medium Concentration SRF) has a fairly soft texture and is slightly difficult to shape, with a less intense green color. Code D (Low Concentration SRF) shows a soft texture and is very difficult to shape.

Analysis of Nitrate and Phosphate Levels
Nutrient content analysis testing was conducted on 300 mL of leachate from each sample, which was analyzed using a UV-Vis Spectrophotometer. The results of the nitrate and phosphate content analysis are presented in Table 1 (refer to the data table from the previous laboratory). The results in Table 1 show that Sample A (Control) has a very high phosphate release compared to fertilizer samples coated with cassava starch and lime peel. In general, Samples A and B showed relatively high nitrate release. Conversely, Sample D showed the lowest nutrient release. The low release in Sample D indicates that the nutrient release rate is the slowest (in line with the slow-release concept).

Table 2 Results of nitrate and phosphate content tests in fertilizer runoff water.

No.	Sample	Nitrate Level (N-NO3-) (ppm)	Phosphate Level (PO4 3-) (ppm)
1.	**A**	15.75	30.88
2.	**B**	15.39	1.25
3.	**C**	3.04	2.21
4.	**D**	1.85	0.56

3.3 Multiple Linear Regression Analysis

$Model\ 1 = N - NO_3^- \sim Cassava + Lime$ peel
$Model\ 2 = P - PO_4^{3-} \sim Cassava + Lime$ peel

Multiple Regression Analysis Results
Model 1 Equation

$$N - NO_3^- = 15.13 - 2.48(Cassava\ starch) + 4.98(Lime\ peel)$$

Each addition of 1 gram of cassava tended to decrease the nitrate concentration by 2.48 ppm. Meanwhile, each addition of 1 gram of lime tended to increase the nitrate concentration by 4.98 ppm

Model 2 equation

$$P - PO_4^{3-} = 29.64 - 2.16(Cassava\ starch) + 0.45(Lime\ peel)$$

Each addition of 1 gram of cassava tends to reduce the phosphate concentration by 2.16 ppm. Meanwhile, each addition of 1 gram of lime tends to reduce the phosphate concentration by 0.45 ppm.

Table 3 Results of SRF Analysis using Multiple Linear Regression.

Input Variable	Effect On $N - NO_3^-$ (Nitrate)	Effect On $P - PO_4^{3-}$ (Phossphate)
Cassava starch	Decreases (-2.48)	Decreases (-2.16)
Lime peel	Increases (+4.98)	Decreases (-0.45)

Discussion
The physical characteristics of the material and product formulation indicate that binder concentration plays a crucial role in the formation of Slow Release Fertilizer (SRF). The lime peel drying process at 50 °C for five days prevents

thermal degradation that can damage bioactive compounds, such as phenolics, flavonoids, pectin, and antioxidants. Based on findings in similar studies (e.g., drying pomegranate peel and seeds), low-to-moderate temperatures (40–60°C) have been shown to be more effective at maintaining antioxidant activity and phenolic compound profiles thanhigh temperatures, which tend to cause a decline in chemical quality and changes in material structure. (Wanderley et al., 2023).

Variation of the physical characteristics of samples A, B, C, and D are influenced by differences in the composition of cassava starch and lime peel powder, which play a role in the gelatinization process and the formation of the SRF matrix. Sample A is the control, in which no cassava starch or lime peel was added. In contrast, samples B, C, and D exhibit different behaviors because cassava starch is able to form a gel when heated with demineralized water. In sample B, the combination of 12 g of starch and 6 g of lime peel powder produced the most moldable and stable texture because this composition created an optimal level of gelatinization where 50 mL of water could be completely absorbed, resulting in a dense, elastic, and not too soft gel structure. This tendency is related to the natural citric acid content in lime peel, which reacts with the hydroxyl groups of starch through a cross-linking esterification mechanism. This interaction produces cross-links that strengthen the gel network, making its mechanical properties more stable and increasing its ability to control the release of phosphate ions (Chamorro et al., 2024)

In samples C and D, changes in the proportion of starch and orange peel shifted the intensity of the cross-links formed. Sample C, which contained 10 g of starch and 3 g of orange peel, had a softer texture than sample B because the amount of citric acid, which acts as a cross-linking agent, was relatively lower, so that a gel network was formed but was not as dense as in sample B. This condition still allows for the absorption of nitrate and phosphate, so that the final nutrient concentration decreases to 3.04 ppm (nitrate) and 2.21 ppm (phosphate). Meanwhile, sample D with 14 g of starch and 4 g of orange peel produced the softest texture because the excess starch caused very strong gelatinization but was not balanced by a sufficient amount of citric acid to form stable cross-links. This softness actually facilitates the formation of larger internal pores, making the adsorption and immobilization of nitrate and phosphate more effective, as evidenced by the lowest final concentrations (1.85 ppm nitrate; 0.56 ppm phosphate). Thus, the relationship between starch content, citric acid concentration from lime peel, and cross-linking mechanisms determines gel structure strength, porosity levels, and the effectiveness of SRF in retaining and reducing nitrate and phosphate release into the growth medium.

Multiple Linear Regression Analysis shows that each component of the formulation has a significant effect on the rate of nutrient release. Each addition of 1 gram of Cassava Starch is consistently correlated with a decrease in Nitrate concentration of 2.48 ppm and a decrease in Phosphate concentration of 2.16 ppm. This proves that Cassava Starch functions as an effective main release inhibitor in reducing the diffusion rate of these two nutrients. Conversely, lime peel showed a contrasting effect; each 1 gram addition correlated with an increase in nitrate concentration of 4.98 ppm, making lime peel the strongest variable facilitating nitrate release. Meanwhile, its effect on phosphate was minimal, causing only a 0.45 ppm decrease in concentration. Based on these findings, it is concluded that Cassava Starch is the main determinant of the slow release effect, while Lime Peel acts as a moderator that accelerates Nitrate release. The success of the SRF formulation is highly dependent on the ratio that ensures the dominance of the inhibiting agent (Cassava Starch) to overcome the acceleration effect caused by lime peel.

Conclusions

The Slow Release Fertilizer (SRF) Sample D formula (14 g cassava starch : 4 g lime peel) proved to be the most effective formula in controlling nutrient release, providing the lowest final nutrient concentrations of 1.85 ppm nitrate and 0.56 ppm phosphate. The success of Sample D lies in achieving an optimal composition ratio, which ensures the dominance of Cassava Starch as the primary inhibiting agent. This is important because regression analysis shows that each addition of Cassava Starch consistently reduces the release of both nutrients, while Lime Peel acts as a counterproductive moderator by significantly increasing Nitrate release (b = +4.98 ppm/g). Although Sample D has the softest texture physically, its superior slow-release performance indicates that efficacy is highly dependent on the dominance of the inhibitor (Cassava Starch) in the coating matrix, which seals the diffusion pathway, overcoming the accelerating effect of Nitrate from Lime Peel.

Conflict of Interest: The authors declare that there is no potential financial or non-financial conflict of interest regarding the research, authorship, and publication of this article

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