

Advanced Nitrogen Management: Effects of Nano-Coated Urea on Nitrogen Leaching in Loam

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ABSTRACT

Urea fertilization using controlled-release techniques is an effective way to reduce nitrogen (N) loss. One technique to decelerate N release and enhance fertilization efficiency involves applying Controlled Release Fertilizer (CRF) via Nano-Coated Urea (NCU) technology. The purpose of this study was to analyze the effect of coated urea application in reducing nitrate leaching rates and improving urea fertilization efficiency in loam soils. The materials used as urea coatings are Bentonite, which has been modified into nanosized particles. The study uses an incubation method with a lysimeter tube and destructive soil sampling to analyze soil nitrate and leached nitrate. The results showed that there was an increase in soil nitrate concentration in the LMC2 treatment (urea coating bentonite) by 28.05% and in the LMC3 treatment (urea coating nano bentonite) by 35.61%, compared to the LMC1 treatment (control urea) of 19.55%.

Keywords: Fertilizer efficiency, lysimeter tubes, nano-technology

INTRODUCTION

Nitrogen is an essential nutrient for plants that is mobile in the soil, so it is easily lost through leaching and evaporation. Nitrogen losses from uncoated urea when applied to crops are prone to evaporation, leaching, and water eutrophication (Uzoh et al., 2019). One of the input materials with a high N content is urea fertilizer. In addition, urea is the most common source of nitrogen, with a N content of around 45–46%, but it is hygroscopic, so it easily evaporates into the air. When urea is applied to soil, the urease enzyme hydrolyzes the urea nitrogen into ammonium (NH_4^+). Ammonium is converted to nitrite (NO_2^-) and then to nitrate (NO_3^-) by the enzyme during nitrification. However, N in urea is easily lost from the soil due to leaching (NO_3^-) if the soil is unable to retain urea because of the large amount of water entering from rain or irrigation (Tina Kogoya, 2018; Hermida & Agustian, 2019).

Various factors, including soil physical and chemical properties, fertilizer type, rainfall intensity, and fertilizer application method, can influence leaching. As such, it can reduce fertilizer efficiency and cause a significant portion of the applied N to be lost to the environment (Grant et al., 2019). Therefore, to increase fertilizer efficiency, it is necessary to improve fertilizer application techniques and modify their physical and chemical properties, such as solubility, shape, and particle size. One innovation to increase fertilization efficiency is the use of Controlled-Release Fertilizer (CRF), which gradually releases nutrients in response to plant needs.

The controlled fertilizer release method involves coating the fertilizer with a semipermeable membrane or melting the nutrients into a matrix that regulates their gradual release (Noor et al., 2022). In previous studies, CRF innovation has not developed fertilizer coating technology with nanomaterials, and the coating materials used were not derived from natural materials, so they cannot

be used as input materials for soil or plants. To enhance innovation, research was conducted on the application of Nano-Coated Urea (NCU) technology, a urea fertilizer coating technique using ZnO and Bentonite minerals to protect the fertilizer from evaporation and increase nutrient release efficiency.

Zhao et al. (2018) reported that Bentonite is effective as a slow-release fertilizer when used as a coating material, with urea release lasting 74 days and increases of 27.52% and 67.97%. The use of Bentonite in research aims to improve soil structure, increase water retention, and reduce nutrient loss. This mineral has a high CEC, so it functions as a slow-release fertilizer. In nano form, Bentonite has a higher surface area and better nutrient-absorption capacity (Rahman, Supriyadi, and Mulyawan, 2022). Whereas ZnO in the study by Umar et al. (2022), which serves as a coating material for urea, has the advantage of releasing higher amounts of Zn (39 and 3.42 mg kg⁻¹) and a relatively long release time (>30 days). Coating urea with ZnO at the nanoscale helps to slow down the release of N until harvest and increase fertilization efficiency. Various studies have shown increased urea efficiency with ZnO coating.

Therefore, the combination of ZnO and bentonite coating is expected to increase the effectiveness of urea fertilizer and reduce nitrate nutrient leaching. This study aimed to analyze and evaluate the effect of urea coating on reducing nitrate leaching and increasing the effectiveness of urea fertilization.

MATERIALS AND METHODS

Time and Location

The research was conducted from June to November 2024. Soil samples for incubation media were collected in Pujon Kidul, Malang, Indonesia, which has a loamy texture and belongs to the Inceptisols soil order. Soil samples were taken at a depth of 0–20 cm (topsoil). The research was conducted at the Soil Laboratory, Faculty of Agriculture, Brawijaya University, and the Applied Physics Laboratory, Faculty of Mathematics and Natural Sciences, Brawijaya University.

Experimental Design

This study used a completely randomized design. The experiment was conducted with five (5) treatments and four (4) replications, as follows: LMC0 = Control Soil; LMC1 = Control Urea; LMC2 = Loam+Urea Coating Bentonite; LMC3 = Loam+Urea Coating Nano Bentonite; LMC4 = Loam+Urea Coating Nano Bentonite and Nano ZnO. The steps in the study will be further explained in the Research Stage section.

This research uses lysimeter tubes to quantify N leaching, with a height of 200 mm and an inner diameter of 35 mm (Figure 1). The soil samples used were Inceptisols, with 100 g of air-dry soil per tube in the topsoil (0–20 cm). The soil was sieved through a 2 mm sieve. The fertilizer was applied during soil preparation. The dosage of each fertilizer used is

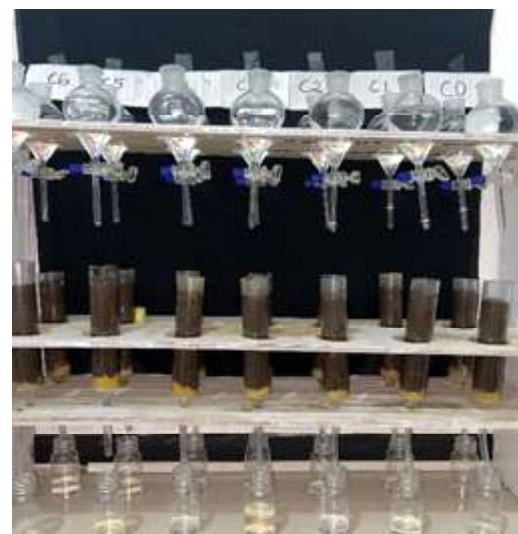
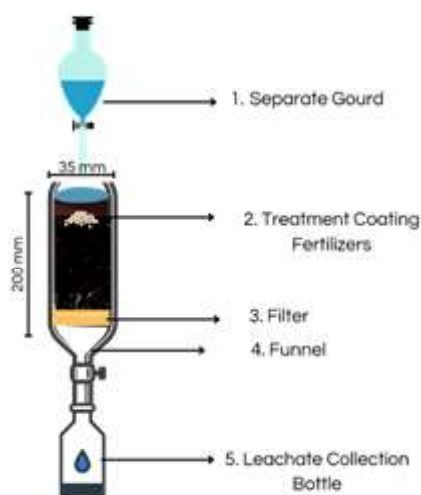


Figure 1. Lysimeter Tube Design.

1.78 grams. Variables observed during the experiment period included Total-N using the Kjeldahl method, Available-N of soil (NH_4^+ and NO_3^-), and Available-N of leachate (NO_3^-) using the Brucin and Spectrofotometry UV VIS method. All analyses are conducted in accordance with the Soil Research Institute’s standard operating procedure. Sampling and observations were conducted at 1, 3, 7, and 12 days after incubation (DAI) for the urea fertilizers.

Research Stages

Preparation and Manufacture of Urea Coating Material

At this stage, the coating material consists of bentonite powder and ZnO, both processed through several stages to produce a nanoscale coating that will be applied to urea fertilizer. The initial stage is the calcination of the material at 400 °C for 6 hours. Calcination is performed to increase the material’s reactivity and stability. Furthermore, the calcined material is sieved to 45 microns, and the material that passes the sieve is weighed to ±15 grams, then reduced in particle size to the nanoscale using a ball mill. The ball milling process is carried out for 6 hours at 400 rpm to ensure a nanoscale particle size, making it more effective as a coating material. After the process is complete, the material is sieved again with the same sieve size to ensure that the resulting particles meet the required nano-size standards. The processed Bentonite and ZnO are then used to make a coating on urea using a Rotary Pan tool.

Entering the urea coating stage, nanosized Bentonite and ZnO are combined with molasses and water as adhesive materials during the coating process. The comparison of the materials used is presented in Table 1. This coating process is carried out using a Rotary Pan tool with a speed of 60–70

rpm, which rotates the urea granules so that the nanomaterial, molasses, and water can be distributed evenly and form a homogeneous layer on each urea granule (Cai & Zhao, 2024; Dissanayake et al., 2024). The total time required is 25 minutes, with several stages and different temperatures at each stage. The first and second 5-minute periods require a temperature of 80–90°C, while the third and fourth 5-minute periods require 50–60°C.

Soil Preparation and Incubation Process

The soil preparation and incubation stages were carried out systematically to ensure homogeneity and accuracy of the results. First, the soil to be used was sieved through a 2 mm (10-mesh) sieve according to the recommended procedure (Medina et al., 2014). After the soil was sieved, the incubation stage began by inserting the sieved soil into a lysimeter tube, a simulation tool that mimics the natural conditions of nutrient leaching in soil. Before adding coated urea fertilizer, the soil was saturated with water to reach field capacity. After the soil was at field capacity, various urea-coated fertilizer treatments, namely Bentonite, ZnO, and a combination of both, were applied to the lysimeter tube at a predetermined coating ratio.

This study was conducted in a laboratory using a nutrient leaching simulation method with a lysimeter tube made of acrylic pipe. The lysimeter tube is designed to facilitate water flow through the soil and collect leachate for analysis. The soil used in the lysimeter tube is 100 grams of air-dried soil. Furthermore, water is added to the soil periodically during the leaching rate simulation, providing 200 mL to simulate nutrient release patterns under rain or irrigation conditions, as reported in the literature (Dubey & Mailapalli, 2019). In this study, the observation time intervals were set at 1, 3, 7, and 12 DAI to examine the release pattern of N from coated

Table 1. Treatment effect on Soil nitrate content with different coatings and days after incubation

Treatment	Soil Nitrate (ppm)			
	1 DAI	3 DAI	7 DAI	12 DAI
LMC0	115.82	96.34a	86.85	32.53
LMC1	157.90	88.26a	104.35	45.46
LMC2	145.62	74.87ab	85.81	58.50
LMC3	155.03	35.23c	77.63	36.06
LMC4	136.46	44.69bc	94.46	36.91
P value	0.10	0.013*	0.95	0.90

Note: DAI: Days After Incubation; (*): significant (p value<0.05). Different letters indicate significant differences among treatments.

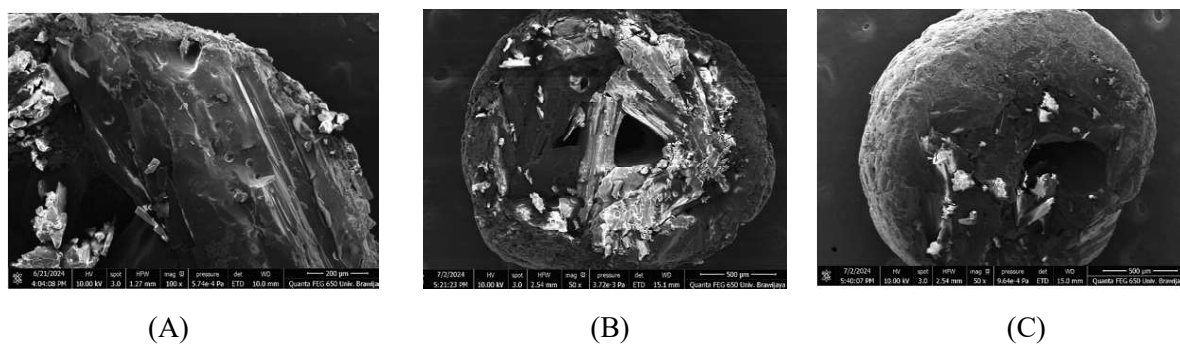


Figure 2. Scanning Electron Microscopy (SEM) of (A) 200 µm urea-coated bentonite; (B) 500 µm urea-coated nano bentonite; (C) 500 µm combination of urea-coated nano ZnO and nano bentonite.

urea fertilizer, with each treatment repeated 4 times. The analysis results were obtained with the following formula:

- Determination of Soil Nitrate using the Brucin method:

$$\text{Nitrogen content (\%)} = \frac{\text{ppm curve} \times \text{mL ekstrak}}{1000 \text{ mL} \times \frac{100}{\text{mg sample}}} \times \text{factor dilution} \times \text{water content factor (1)}$$

- Determination of Water Nitrate Using Spectrofotometry UV VIS Method:

$$\text{Water Nitrate content (m.e.:1)} = \frac{\text{ppm curve}}{\text{bst N}} \times \text{factor dilution (2)}$$

Research Data Analysis

The data was analyzed by analysis of variance (ANOVA) and further tested by LSD (Least Significant Difference) analysis with the F test at the 5% level if the data was significantly different. The test used the R Studio application.

RESULTS AND DISCUSSION

SEM Analysis of Nano-Coated Urea

The coated urea was characterised using SEM (Scanning Electron Microscopy) to determine the morphology of the fertilizer coating with uncoated urea. In this case, the term ‘coated urea’ refers to the shape and structure of a coating material (Uzoh et al., 2019). The coated fertilizer is urea coated with the minerals Bentonite and ZnO, which have been converted to powder. Coated urea has hydrophilic properties, meaning the urea coating material can increase water retention and N adsorption, thereby maximising N supply and delaying leaching (Ma et al., 2019; Shetty et al., 2024). However, this research experiment has shortcomings, especially the uneven, non-uniform morphology of the urea coating thickness. Figure 2

shows the results of SEM readings at magnifications of 200 µm and 500 µm. A study on coating materials to control fertiliser release found that polymeric materials with thicknesses of 50-100 µm were effective in controlling N release over several weeks to months. The study showed that higher crosslinking densities led to slower release rates (Vermoesen et al., 2023). Therefore, in this research experiment, one limitation is the thickness of the coating material, which is not ideal.

Nitrate (NO_3^-) Leaching in Soil

The results of the ANOVA for the leaching parameters of soil nitrate (Table 1) and water nitrate (Table 2) are presented.

Nitrate Leachate (NO_3^-)

The results of the ANOVA for soil nitrate (Table 1) showed that at 3 DAI, there was a significant difference, with the highest value in treatment LMC0 (control soil). However, the treatment was

Table 2. Treatment effect on Leachate nitrate content with different coatings and days after incubation.

Treatment	Water Nitrate (m.e/L)			
	1 DAI	3 DAI	7 DAI	12 DAI
LMC0	0.77	0.17	0.36	0.15
LMC1	0.80	0.19	0.45	0.17
LMC2	0.53	0.30	0.30	0.19
LMC3	0.67	0.20	0.28	0.18
LMC4	0.58	0.39	0.32	0.34
P value	0.60	0.27	0.16	0.33

Note: DAI: Days After Incubation; (*): significant (p value<0.05). Different letters indicate significant differences among treatments.

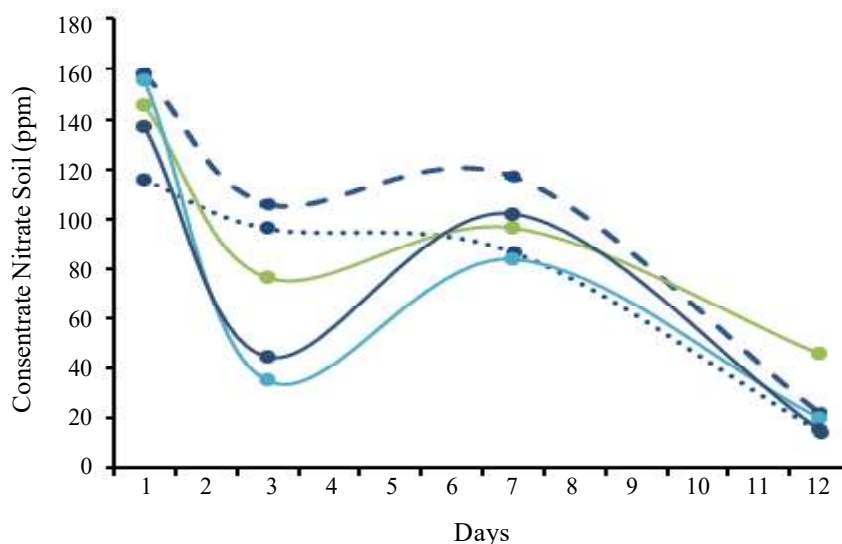


Figure 3. Soil Nitrate. : LMC0, - - : LMC1, — : LMC2, — : LMC3, — : LMC4.

not significantly different from LMC1 or LMC2 (control urea and urea-coated Bentonite). In addition, this treatment was significantly different from LMC3 and LMC4 (urea-coating nano-bentonite and urea-coating combination Nano-bentonite & Nano ZnO). Furthermore, the ANOVA results at 7 and 12 DAI were not significant across any treatment. Several studies show that Bentonite is a montmorillonite clay mineral (2:1 type) that can bind multiple cations. Bentonite is also known to have a higher CEC, which can act as a slow-release fertilizer (Paskovi  et al., 2018). The ANOVA on water nitrate (Table 2) showed that the overall treatment effect across observation times was not significant. The insignificant difference between treatments and observation times is assumed to reflect a low nitrate leaching rate and thus does not have a real effect on the treatment.

Nitrate (NO₃⁻) Release Pattern in Soil and Water

The graph of soil nitrate concentration (NO₃⁻) (Figure 3) at observation times 1 to 12 DAI shows significant fluctuations in several urea coating treatments. As is known at 1 DAI, the nitrate concentration in each treatment is relatively high. However, there is a decrease in the graph at 3 DAI in all treatments. Interestingly, although all treatments showed a significant decrease at 3 DAI, a peak at 7 DAI was followed by another decrease until 12 DAI during the incubation period. The decrease in nitrate concentration at 3 DAI is explained in the study by Swify, Ma eika, and Volungevi us (2023), which reports that nitrate (NO₃⁻) accumulation increases 1–2 weeks after

application. In contrast, at 1–4 DAI, ammonium (NH₄⁺) concentration increases. At that time, there will also be a decrease due to denitrification, which converts nitrate to nitrogen gas.

The graph for 7 DAI shows a peak increase in nitrate concentration for all urea treatments, except for the soil control treatment, which continued to decline until 12 DAI. The LMC3 treatment (urea-coated nano bentonite) had the lowest nitrate concentration compared to the coating treatment. Due to its high CEC and large surface area, modified nano-bentonite can effectively retain N ions in the root zone. It can enhance nutrient uptake, reduce nitrate leaching, and ensure that N remains accessible to plant roots (Messiga et al., 2020). Furthermore, since 2 DAI to 7 DAI were followed by a decrease in NH₄⁺, indicating that N-ammonium was nitrified to nitrate, the process starts at a very fast rate. Therefore, over time, N content in the soil (NH₄⁺ and NO₃⁻) may fluctuate. Although the release rate may not correlate linearly with time, this is because the process runs according to the plant’s nutrient requirements and absorption process (Marchetti & Castelli, 2011; Swify et al., 2022).

The results of soil nitrate leaching are shown in the graph (Figure 4), which indicates that on 1 DAI, the LMC1 treatment (urea control) had the highest leaching concentration, followed closely by the LMC0 treatment (control). However, all the urea coating treatments also leached. Meanwhile, from 3 DAI to 12 DAI, the LMC3 treatment (urea-coated Nano bentonite) showed a significant decrease and became the treatment with the lowest leaching concentration. In comparison, the LMC4 treatment had a relatively stable decrease from 1 DAI to 12 DAI. It may be due to the Nano coating, which

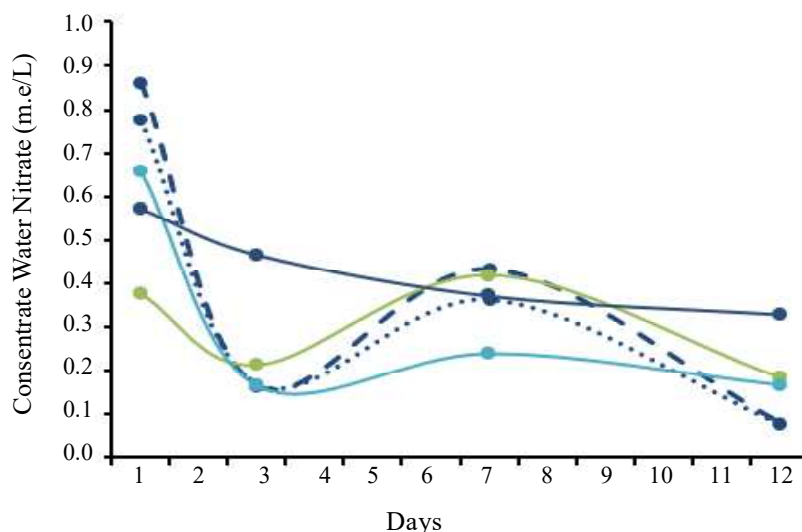


Figure 4. Nitrogen Soil Leaching. ...●... : LMC0, -●- : LMC1, —●— : LMC2, —●— : LMC3, —●— : LMC4.

allows N to be released gradually, keeping the concentration of available N in the soil stable. The mixture of Bentonite with various N sources has been proven to be effective in reducing nitrate leaching from the soil (Hussain et al., 2022).

CONCLUSIONS

Based on the results of the study, coated urea shows a significant difference only at 3 DAI in soil nitrate, but not in water nitrate. However, the leaching rate of nitrate in water decreases from 1 DAI to 12 DAI compared to the control treatment. It is supported by the fact that the coating material can slow the rate of nitrate release from soil and reduce nitrate leaching. However, the effectiveness of this fertilizer required testing over a longer period to determine its long-term nitrate release pattern.

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