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# Changes in Chemical Properties of Sipramin-Affected Paddy Soil during Incubation with Humic Acid and Nitrogen Fertilizer

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## Abstract

Continuous use of Sipramin fertilizer can lead to sodium ($Na^+$) accumulation in the soil. Sodium accumulation damages soil physical properties, which also affects the chemical quality and nutrient supply. This study aimed to evaluate the effects of various doses of humic acid and nitrogen fertilizer sources on soil chemical properties. This study was arranged in a factorial completely randomized design (CRD) with 2 factors: humic acid doses (0, 20, 40, and 60 kg ha$^{-1}$) and nitrogen fertilizer types (control, NPK Phonska, Urea, $KNO_3$, and MAP) with a recommended rate of 92 kg ha$^{-1}$. The study consisted of 20 treatment combinations with 3 replications, yielding a total of 60 experimental units arranged randomly. Soil samples were collected at 7 and 21 days after treatment (DAT) to analyze pH, organic C, cation exchange capacity (CEC), exchangeable Na, and available N ($NH_4^+$ and $NO_3^-$). The results showed that the interaction between humic acid and nitrogen fertilizer did not significantly affect the chemical properties of the Sipramin-affected soil. In general, the effects of both treatments depended on the parameters and the time of observation. At 7 DAT, several variables showed significant effects, whereas at 21 DAT, some of those effects were no longer significant.

**Keywords:** chemical properties; nitrogen fertilizer; paddy soil; Sipramin; soil amendment

## INTRODUCTION

The paddy fields of Winongo Village, Manguharjo Sub-district, Madiun City, are suspected to be affected by Sipramin. Sipramin is a liquid organic material derived as a by-product of seasoning production made from sugarcane molasses (Bangun and Suryanto, 2020). Amine-type Sipramin has been applied for almost 10 years and has become a staple organic fertilizer for farmers. However, based on exploration and interviews, rice productivity has actually declined. Research by Suntoro et al. (2012) reported that amine-type Sipramin has a pH of 5.48 with a total N content of 4.56%, total P of 0.044%, total K of 1.30%, Na of 0.75 me%, and relatively low levels of several heavy metal contents. Based on the results of initial soil analysis, the exchangeable sodium ($Na^+$) value was 1.10 cmol(+) kg$^{-1}$, which is classified as very high. Based on this data, it is suspected that continuous use of Sipramin fertilizer can lead to $Na^+$ accumulation in the soil.

The accumulation of $Na^+$ from Sipramin has a major impact on the deterioration of soil physical properties, characterized by crust formation and compaction due to soil dispersion on soil porosity. These conditions affect microorganism activity in the soil, thereby influencing nutrient availability and their uptake by plant roots, including nitrogen. Nitrogen is a primary macronutrient that plays an important

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role in plant growth and development, especially during the vegetative phase (Iswiyanto et al., 2023). Plants use it for protein synthesis, which serves as a component of plant organs. Therefore, appropriate land management efforts are needed, including the application of soil amendments and suitable nitrogen fertilization to reduce the impact of $Na^+$ from Sipramin and increase nitrogen availability in the soil. One soil amendment that can serve as an alternative solution to this problem is humic acid.

Humic acid is an organic colloid formed from the decomposition of organic matter that has undergone humification and is soluble in alkali (Hidayat et al., 2023). The use of humic acid can increase cation exchange capacity (CEC), organic C, soil pH, and enlarge the surface area of soil colloids. Humic acid can interact with metal ions, oxides, minerals, and organic compounds through its functional groups (Male et al., 2022). The negatively charged groups of humic acid can interact with positively charged ions through complex bonding, adsorption, and chelation. Therefore, humic acid is considered capable of reducing excess sodium in the soil. Humic acid can be used as a soil amendment and nitrogen fertilizer companion that can improve soil physical, chemical, and biological properties, increase nitrogen fertilization efficiency, and overcome nitrogen deficiency in the soil. In addition, organic matter and soil conditioners such as humic acid can affect bacterial activity, structural improvement, carbon supply, and nutrient cycling (Maffia et al., 2025).

Nitrogen fertilizers are not only important as macronutrients for plant growth, but also play a key role in modifying soil chemical properties. Ammonium-based nitrogen fertilizers can affect soil pH through nitrification, which releases hydrogen ions ($H^+$) and leads to soil acidification (Kusumawardana et al., 2025). Conversely, nitrate-based nitrogen fertilizers can contribute to the leaching of base cations, thereby affecting soil chemical balance. Nitrogen dynamics in the soil, including mineralization, immobilization, nitrification, and denitrification, are closely linked to changes in soil chemical properties. Therefore, understanding the role of nitrogen fertilizers as a factor influencing soil chemical properties is crucial for evaluating their combined effects with humic acids. These humic acid compounds can enhance fertilization efficiency by reducing nutrient losses through leaching and increasing their availability to plants (Nuraini and Zahro, 2020). Additionally, humic acid acts as a soil conditioner and biostimulant that can improve soil structure, increase CEC, and support soil microorganism activity, thereby enhancing overall soil fertility (Rahmayuni et al., 2025).

The combined application of humic acid and nitrogen fertilizer can increase nitrogen uptake in rice plants (Nuraini and Zahro, 2020). As in the research by Manjeera et al. (2021), the application of 30 kg $ha^{-1}$ humic acid combined with the 100% recommended nitrogen dose (120 kg N $ha^{-1}$) provided the highest soil available nitrogen. Based on research by Mindari et al. (2025), the application of 1 ton $ha^{-1}$ biosilica and 40 kg $ha^{-1}$ humic acid is the best combination capable of increasing organic C, total N, exchangeable K, and soil pH. Based on the background of the problems above, further research is needed to examine the effects of various doses of humic acid and nitrogen fertilizer sources on soil chemical properties and nitrogen availability in Sipramin-affected soils.

## MATERIALS AND METHOD

### Time and location

This research was conducted from February to August 2025. Soil samples were collected from paddy fields in Winongo Village, Manguharjo Sub-district, Madiun City, East Java. Planting activities were carried out in the greenhouse of the Faculty of Agriculture, Universitas Pembangunan Nasional Veteran Jawa Timur, while laboratory analyses were conducted at the Land Resources Laboratory of the same faculty.

### Research design

The research was arranged in a factorial completely randomized design (CRD) consisting of 2 factors. The first factor was 4 doses of humic acid (H): H0 = 0 kg $ha^{-1}$ (Control), H1 = 20 kg $ha^{-1}$ (0.0335 g 3.35 $kg^{-1}$ soil), H2 = 40 kg $ha^{-1}$ (0.067 g 3.35 $kg^{-1}$ soil), and H3 = 60 kg $ha^{-1}$ (0.1005 g 3.35 $kg^{-1}$ soil). The second factor was 5 types of nitrogen fertilizer with a nutrient amount of 92 kg N $ha^{-1}$ (Jauhari et al., 2021), consisting of N0 = no fertilizer application (control), N1 = NPK Phonska, N2 = Urea, N3 = $KNO_3$, and N4 = MAP. The study consisted of 20 treatment combinations with 3 replications, yielding a total of 60 experimental units arranged randomly.

## Research implementation stages

*Soil media preparation*

Soil media preparation involved drying the soil, then grinding and sieving it through a 2 mm sieve to ensure uniform soil particle size. The soil was then placed into buckets with uniform weight, namely 3 kg of oven-dry soil equivalent to 3.35 kg of air-dry soil. The results of the initial soil characterization analysis showed a pH of 6.39 (slightly acidic), organic C of 2.90% (moderate), CEC of 39.79 cmol(+) $kg^{-1}$ (high), exchangeable Na of 1.10 cmol(+) $kg^{-1}$ (very high), $NH_4^+$ of 239.55 ppm, and $NO_3^-$ of 31.94 ppm.

*Soil amendment and fertilizer incubation*

The soil amendment used was humic acid (HumaTOP brand) produced by PT. Prima Agro Tech from natural leonardite rock extraction. The humic acid was dissolved in water according to the treatment doses and applied by watering evenly under field capacity conditions. The fertilizers applied were NPK Phonska, Urea, $KNO_3$, and MAP with a recommended dose of 92 kg N $ha^{-1}$ (Jauhari et al., 2021). This fertilizer application was carried out simultaneously with the soil amendment and then incubated to react with the soil.

*Soil sample collection*

Soil samples for laboratory analysis were collected at 3 points to represent the sample conditions of each treatment. Sampling was conducted twice, at 7 and 21 days after treatment (DAT).

## Soil analysis parameters

The soil analysis parameters included pH, measured using the potentiometric method; organic C using the Walkley and Black method; exchangeable Na using the 1 N ammonium acetate extraction method at pH 7; CEC using the same extraction method, and available N using the titrimetric distillation method. Soil sample analysis methods were based on the Guidelines for Soil, Plant, Water, and Fertilizer Analysis from the Soil Research Institute (Eviati et al., 2023).

## Data analysis

The research data were analyzed using analysis of variance (ANOVA) at a 5% significance level to determine the effects of the treatments. If there were significant differences among the treatments, an Honestly Significant Difference (HSD) test was performed at the 5% significance level to identify the best-performing treatments. The statistical software used for data analysis in this research was Microsoft Excel and Minitab.

# RESULTS AND DISCUSSION

Soil with high sodium content can lead to a decline in the quality of its physical, chemical, and biological properties. This condition affects agricultural productivity and the sustainability of production systems. Based on soil analysis results after treatment, changes in soil chemical properties varied depending on the parameter and observation time, with some variables showing significant effects at 7 or 21 DAT, while others were not significantly affected. The research results of soil chemical property changes at 7 DAT are shown in Table 1, whereas those at 21 DAT are shown in Table 2.

## Soil pH

Based on the results of ANOVA, at 7 DAT, the single factor of nitrogen fertilizer had a significant effect on soil pH ($p < 0.05$), while the single factor of humic acid and the interaction between 2 factors did not show a significant effect ($p > 0.05$). The pH values under nitrogen fertilizer treatments ranged from 6.01 to 6.25, indicating slightly acidic conditions. The N0 treatment produced the highest result of 6.25, which was significantly different from the N1 treatment with a pH of 6.01 (Table 1). Based on the soil pH data in Table 1, the application of nitrogen fertilizer decreased soil pH, where NPK Phonska fertilizer (N1) caused the greatest decrease from pH 6.39 (baseline soil) to 6.01. According to Kaya (2013) and Kusumawardana et al. (2025), NPK fertilizer contains sulfur and ammonium, which are hydrolyzed to produce $H^+$, thus lowering soil pH.

Based on the results of ANOVA, the single factor of humic acid had a significant effect on soil pH ($p < 0.05$) at 21 DAT, while the single factor of nitrogen fertilizer and the interaction between 2 factors did not show a significant effect ($p > 0.05$). The pH value of the H2 treatment at 21 DAT showed the highest value of 6.59, which was classified as slightly acidic (Table 2). Soil pH tended to increase with humic acid application compared to the control (no humic acid) across different nitrogen fertilizer levels. This indicates that humic acid had begun to react in the soil and could increase soil pH through buffering capacity. Humic acid has the ability to act as a buffer that

Table 1. Soil chemical properties at 7 days after humic acid and nitrogen fertilizer treatment

| Treatment | 7 DAT | | | | | |
|---|---|---|---|---|---|---|
| | pH | Organic C | CEC | Exch-Na | $NH_4^+$ | $NO_3^-$ |
| | | % | Cmol(+) $kg^{-1}$ | | ppm | |
| Humic acid | | | | | | |
| H0 | 6.13 | 2.69ab | 44.69 | 1.85c | 314.69 | 46.88a |
| H1 | 6.08 | 2.74b | 42.30 | 1.51b | 344.24 | 84.88b |
| H2 | 6.10 | 2.63a | 40.61 | 1.27a | 367.99 | 78.76b |
| H3 | 6.06 | 2.65a | 41.90 | 1.56b | 336.04 | 60.03ab |
| Tukey's HSD 5% | ns | 0.08 | ns | 0.22 | ns | 29.77 |
| Nitrogen fertilizer | | | | | | |
| N0 | 6.25b | 2.75b | 41.02 | 1.51a | 338.77 | 62.87 |
| N1 | 6.01a | 2.65a | 42.50 | 1.79b | 337.35 | 70.02 |
| N2 | 6.10ab | 2.66ab | 45.08 | 1.47a | 348.04 | 67.63 |
| N3 | 6.10ab | 2.66ab | 41.23 | 1.51a | 348.87 | 63.29 |
| N4 | 6.01a | 2.67ab | 42.04 | 1.46a | 330.65 | 74.38 |
| Tukey's HSD 5% | 0.15 | 0.10 | ns | 0.26 | ns | ns |

Note: Numbers followed by the same letter in the same column indicate results that are not significantly different at the 5% HSD level; ns is not significant

can absorb or release $H^+$ to stabilize soil pH. According to Mohammed et al. (2025), the mechanism by which humic acid affects soil pH is controlled by the presence of active functional groups, such as carboxyl and phenolic groups, which act as weak acids through a buffering mechanism.

**Soil organic C**

At 7 DAT, the single factors of humic acid and nitrogen fertilizer significantly affected soil organic C ($p < 0.05$), while the interaction between the 2 factors did not show a significant effect ($p > 0.05$). This indicates that each factor worked independently in influencing soil organic C during the early growth phase. Table 1 shows that the H1 treatment produced the highest organic C value of 2.74% with moderate criteria. This value was significantly different from the H2 and H3 treatments (Table 1), but not significantly different from the H0 treatment. This indicates that within a 7-day period, the decomposition process of humic acid had not shown a significant effect on the increase of soil organic C. Nuryani et al. (2003) and Chairunnisya et al. (2017) stated that organic matter applied to the soil will undergo a decomposition process that can increase soil carbon and organic acid content.

The N0 treatment at 7 DAT produced the highest organic C result of 2.75% with moderate criteria, which was significantly different from the N1 treatment. The nitrogen fertilizer used was inorganic; therefore, it did not directly contribute organic matter to the soil. The application of nitrogen fertilizer can also increase microbial activity and carbon mineralization; thus, the treatment without fertilizer yielded the highest organic C result. As stated by Setiawati (2014), the continuous use of inorganic fertilizers will increase the mineralization of soil organic matter, leading to a decrease in soil organic C.

Based on the results of ANOVA, neither humic acid nor nitrogen fertilizer, nor the interaction factor of both, had a significant effect on soil organic C at 21 DAT ($p > 0.05$). Although organic C values at 21 DAT were numerically higher than those at 7 DAT, the difference was not statistically significant (Table 2). This indicates that humic acid acts as an effective agent in increasing soil organic C through direct carbon contribution. Nuraini and Zahro (2020) stated that humic acid is a humified fraction rich in carbon; therefore, it can supply organic C to the soil.

**Soil CEC**

CEC is the amount of cations that can be adsorbed or exchanged by the soil, expressed in units of cmol(+) $kg^{-1}$ (Syachroni, 2019). Clay and organic matter contribute to soil CEC because they have negative charges that can attract positively charged cations. Based on the results of ANOVA, neither humic acid nor nitrogen fertilizer, nor their interaction, had a significant effect on CEC at 7 DAT ($p > 0.05$). Table 1 shows that the H0 treatment had the highest CEC value compared to the humic acid application

Table 2. Soil chemical properties at 21 days after humic acid and nitrogen fertilizer treatment

| Treatment | 21 DAT | | | | | |
|---|---|---|---|---|---|---|
| | pH | Organic C | CEC | Exch-Na | $NH_4^+$ | $NO_3^-$ |
| | | % | Cmol(+) kg$^{-1}$ | | ppm | |
| Humic acid | | | | | | |
| H0 | 6.30$^{a}$ | 2.81 | 45.96$^{ab}$ | 0.98$^{a}$ | 359.60 | 40.24 |
| H1 | 6.52$^{b}$ | 2.79 | 44.21$^{a}$ | 1.39$^{c}$ | 331.55 | 65.73 |
| H2 | 6.59$^{b}$ | 2.77 | 43.05$^{a}$ | 1.24$^{b}$ | 332.69 | 62.48 |
| H3 | 6.57$^{b}$ | 2.79 | 49.89$^{b}$ | 1.26$^{b}$ | 303.47 | 61.41 |
| Tukey's HSD 5% | 0.16 | ns | 4.85 | 0.10 | ns | ns |
| Nitrogen fertilizer | | | | | | |
| N0 | 6.53 | 2.81 | 43.54 | 1.26$^{ab}$ | 372.44 | 43.38 |
| N1 | 6.47 | 2.76 | 46.19 | 1.19$^{ab}$ | 316.56 | 68.50 |
| N2 | 6.54 | 2.80 | 45.54 | 1.16$^{a}$ | 360.68 | 52.40 |
| N3 | 6.55 | 2.81 | 44.83 | 1.19$^{ab}$ | 299.54 | 55.74 |
| N4 | 6.39 | 2.76 | 48.80 | 1.29$^{b}$ | 309.91 | 67.32 |
| Tukey's HSD 5% | ns | ns | ns | 0.12 | ns | ns |

Note: Numbers followed by the same letter in the same column indicate results that are not significantly different at the 5% HSD level; ns is not significant

treatments. This is presumably because humic acid requires sufficient time to react in the soil, as supported by the organic C data at 7 DAT, which has not yet shown a significant effect on humic acid treatment. Organic C content is an indicator of soil organic matter that affects the soil CEC. Salawati et al. (2022) stated that organic matter has a higher adsorption capacity than clay colloids; therefore, the higher the soil organic matter content, the higher the CEC values.

Based on the results of ANOVA, the single factor of humic acid had a significant effect on soil CEC ($p$ < 0.05), while the single factor of nitrogen fertilizer and their interaction did not show a significant effect ($p$ > 0.05). Table 2 shows that the H3 treatment gave the highest CEC value of 49.89 cmol(+) kg$^{-1}$, with very high criteria, which was significantly different from the H2 treatment. This indicates that the application of humic acid during that period began to increase soil CEC. The application of humic acid as a soil amendment can increase soil CEC value through the binding of organic compound chains with nutrients in the soil. Humic acid has active functional groups that can dissociate $H^+$ ions in the soil solution, producing negatively charged groups (-COO- and -O-) that function as cation exchange sites (Ampong et al., 2022). As stated by Aprilia et al. (2024), humic fractions have very high CEC due to the negative charge from $H^+$ ion dissociation of various functional groups, thereby enhancing the soil's ability to bind, adsorb, and exchange cations in the soil.

**Soil exchangeable Na**

Exchangeable Na is the amount of sodium ions present on the surface of soil particles that can be released for plant use or replaced by other cations. Sodium is not an essential nutrient, therefore low sodium concentrations are generally beneficial. Jin et al. (2024) stated that excessive $Na^+$ in the soil can affect nutrient availability and absorption through direct and indirect ion competition, by reducing the mass flow of nutrients to the roots due to increased osmotic pressure in the soil solution. High sodium concentrations can also damage soil structure, causing compaction (Gyhan and Rauf, 2023). According to Choudhary and Kharche (2018), the accumulation of $Na^+$ in the soil also reduces the content of multivalent ions. It increases the percentage of sodium in soil colloids, which can break down soil aggregates into individual particles and increase soil dispersion.

Based on the ANOVA results, the single factors of humic acid, nitrogen fertilizer, and their interaction had a significant effect on exchangeable Na at 7 DAT ($p$ > 0.05). Table 1 shows that the H2 treatment resulted in the lowest exchangeable Na content at 7 DAT, with a value of 1.27 cmol(+) kg$^{-1}$ compared to without humic acid treatment. Based on the interaction results, the H2N2 treatment (40 kg ha$^{-1}$ humic acid + urea) provided the lowest exchangeable Na in 7 DAT at 1.01 cmol(+) $kg^{-1}$. This is presumably because humic acid acts as a chelating agent capable of binding metal ions or cations in the soil, including sodium. According to Nasution et al. (2024), humic acid is capable of binding metal ions or cations on the surface of soil minerals so that these metal ions or cations become insoluble in the soil.

Exchangeable Na is the amount of sodium ions that are on the surface of soil particles and can be released for plant use or replaced by other cations. High soil sodium concentration can increase the osmotic value, causing plasmolysis and disrupting plant growth. High sodium concentration can also damage soil structure, leading to compaction (Gyhan and Rauf, 2023). Table 2 shows a decrease in exchangeable Na content in all treatments. This reduction is presumably due to adsorption activity by the functional groups of humic acid. As stated by Al-Falahi et al. (2022), humic acid contains functional groups such as carboxylate and hydroxyl groups that play a role in chelation, sodium ion adsorption, and the formation of soluble and mobile organic complexes, thereby increasing the possibility of sodium ion leaching in soil.

**Soil available N**

The ANOVA results showed that the combination treatment of humic acid and nitrogen fertilizer did not have a significant effect on nitrogen availability at 7 and 21 DAT, either as $NH_4^+$ or $NO_3^-$ ($p > 0.05$). However, humic acid treatment significantly affects the levels of $NO_3^-$ in the soil ($p < 0.05$). Table 1 shows that the H2 treatment resulted in the highest $NH_4^+$ concentration of 367.99 ppm with 78.76 ppm $NO_3^-$, while the H1 treatment produced the highest $NO_3^-$ concentration of 84.88 ppm with 344.24 ppm $NH_4^+$, which was not significantly different from the H2 and H3 treatments. The H0 treatment resulted in the lowest $NH_4^+$ and $NO_3^-$ concentrations of 314.69 and 46.88 ppm, respectively. Humic acid did not show a significant difference but tended to maintain the availability of $NH_4^+$ and $NO_3^-$ compared with treatments without humic acid application. According to Radite and Simanjuntak (2020), humic acid can adsorb nutrients, thereby maintaining nutrient availability in soil and optimizing N fertilization.

The N3 treatment at 7 DAT resulted in the highest $NH_4^+$ content of 348.87 ppm with 63.29 ppm $NO_3^-$, while the N4 treatment produced the highest $NO_3^-$ content of 74.38 ppm with 330.65 ppm $NH_4^+$. $KNO_3$ is a nitrate source, but the high ammonium content in this treatment is presumably due to soil microorganism activity that reduces $NO_3^-$ to $NH_4^+$. Conversely, MAP contains nitrogen in the form of ammonium but produced the highest nitrate content, presumably due to nitrifying microbial activity supported by humic acid and the phosphorus content in MAP as an energy source. This indicates that nitrogen transformation in the soil is not only determined by the form of nitrogen input but is also influenced by microbiological activity and organic carbon availability.

Table 2 shows that $NH_4^+$ and $NO_3^-$ contents decreased; however, $NH_4^+$ showed higher results compared to $NO_3^-$. This decrease is presumably due to the nitrogen mineralization process that occurs dynamically and begins to be absorbed by rice plants for growth. The $NH_4^+$ content in the soil was higher than the $NO_3^-$ content in all treatments. This was due to waterlogged soil conditions that inhibited mineralization. Wu et al. (2024) reported that anaerobic conditions in waterlogged soil in paddy fields limited nitrification and increased denitrification rates, resulting in a greater dominance of $NH_4^+$ and its direct uptake by rice plants. The low nitrate content can also be attributed to its high solubility and mobility in soil, making it susceptible to leaching with water flow. In contrast, ammonium is bound to soil particles, making it more stable and not easily leached. According to Hermanto et al. (2012) and Nuraini and Zahro (2020), nitrogen fertilization combined with humic acid can increase nitrogen availability through the role of humic acid in slowing down the release of nitrogen into $NO_3^-$.

## CONCLUSIONS

The interaction between humic acid and nitrogen fertilizer did not have a significant effect on the chemical properties of soil affected by Sipramin. In general, the effects of both treatments depended on the parameters and the time of observation. At 7 DAT, humic acid had a significant effect on organic C, exchangeable Na, and $NO_3^-$, while nitrogen fertilizer had a significant effect on pH, organic C, and exchangeable Na. Meanwhile, at 21 DAT, humic acid had significant effects on pH, CEC, and exchangeable Na, whereas nitrogen fertilizer had a significant effect only on exchangeable Na.

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