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Synthesis of Struvite Fertilizer ($MgNH_4PO_4.6H_2O$) from Cow Urine and Bittern by Precipitation Technique

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Abstract

The need for fertilizers has significantly increased due to the expanding global population and the mounting pressure on agricultural systems to increase food production. Fertilizers are essential in upgrading soil fertility and increasing crop productivity, making them indispensable in contemporary agriculture. Extensive usage of chemical fertilizers has led to environmental issues such as soil deterioration, water contamination, and the exhaustion of non-renewable resources like phosphate rock. Phosphorus, one of the essential macronutrients for plants, is a critical component of commercial fertilizers. Struvite, with its high phosphate content, presents a viable alternative to conventional fertilizers. Struvite ($MgNH_4PO_4{\cdot}6H_2O$) is a crystalline compound comprising magnesium, ammonium, and phosphate. Cow urine, which contains nitrogen, phosphate, and potassium, can serve as a nutrient source for plants. In the context of fertilizer production, bittern serves as a source of magnesium. This study investigated the production of struvite from cow urine and bittern using a precipitation technique. The study aimed to determine the optimal conditions for maximizing struvite production, focusing on the ratio of cow urine to bittern and the storage duration of cow urine. The research involved the preparation and characterization of raw materials, struvite synthesis, and the analysis of struvite characteristics. The optimal conditions were found to be a molar ratio of cow urine to bittern of 1:1 and a cow urine storage time of one week. UV-VIS spectrophotometry was utilized to analyze phosphate and ammonia contents, yielding optimal values of 72.417 mg/kg and 0.715 mg/kg, respectively. XRF analysis indicated a magnesium content of 26.000 mg/kg. SEM and XRD analyses revealed that the morphology of the struvite was orthorhombic at the optimum conditions. FTIR analysis was used to identify N-H, P-O, and water vibrations.

Keywords: alternative phosphate, cow urine, bittern, precipitation technique, struvite

1. Introduction

Intensive farming practices intensify the demand for fertilizers to meet the requirements of human existence. Phosphorus is an essential ingredient for plants. Phosphorus is categorized as a macronutrient that exerts beneficial effects on the stimulation of plant root development and the acceleration of plant maturation. The main source of phosphorus in the production of commercial phosphate fertilizers, such as NPK fertilizers, is derived from the mining of phosphate rocks. The world's primary phosphorus producers are China, Morocco, Western Sahara, the United States, Russia, Jordan, Saudi Arabia, Brazil, Egypt, Vietnam, and Tunisia. Phosphorus-producing phosphate rock is reported to have decreased, and natural phosphate reserves of various qualities worldwide are estimated at 200-300 billion tons (Musaad, 2018).

One of the countries that imports phosphate rock as raw material for the fertilizer industry is Indonesia. In anticipating the scarcity of phosphate sources, it is necessary to innovate to ensure food security in the future. Some known phosphate sources are leachate from landfills and wastewater treated aerobically and anaerobically (Suprihatin. et al., 2019). Other potential phosphates are laundry waste (Pungut et al., 2021) and animal urine (Krishnamoorthy et al., 2021). Cow urine is a

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potential source of phosphorus and ammonia with a nutrient content of 100 ppm and 430 ppm, respectively (Edahwati et al., 2020b). Putra et al. (2022) state that one cow produces as much as 8 liters of daily urine. It is dumped directly into the farm or ditch, causing environmental pollution and unpleasant odors. Cow urine can be used as a raw material for plant fertilizer, namely struvite (Krishnamoorthy et al., 2021).

Struvite ($MgNH_4PO_4.6H_2O$) is a crystalline mineral substance containing magnesium, ammonium, and phosphate. Struvite can be used as fertilizer, providing phosphorus and magnesium nutrients. The advantage of struvite as a fertilizer is slow release so that the main content of struvite can last long in the soil and plants can take advantage of the nutrient significantly (Edahwati et al., 2021). The precipitation technique is the most commonly used technique for synthesizing struvite. Struvite precipitation is a process formed by a chemical reaction to form a precipitate with the help of the precipitating elements Mg^{2+}, NH_4^+, and PO_4^{3-}. The purpose of struvite precipitation is to reduce the concentration of ammonia and phosphate so that they will form a precipitate in the form of struvite crystals. The factors that influence this process include the chemical content of wastewater (degree of saturation with magnesium, ammonium, and phosphate; the presence of other ions; ionic strength); temperature; and the most influential is the molar ratio of Mg^{2+}, NH_4^+, and PO_4^{3-}, and pH (Suprihatin et al., 2019).

Based on data from the (Badan Pusat Statistik, 2022), the cattle population in Indonesia is more than 18 million heads, and Aceh is one of the top ten provinces in cattle farming, with a total of 400 thousand cows. The synthesis of struvite from cow urine is promising because it already contains high ammonium and phosphate content (Krishnamoorthy et al., 2021). The only primary additive needed to produce struvite from cow urine is magnesium. Previous research has tried to utilize cow urine in struvite by adding commercial chemicals such as $MgCl_2$ (Edahwati et al., 2020b).

One of the potential economic sources of magnesium can be obtained from bittern. Bittern is a concentrated liquid made from the residual salt water obtained from the salt crystallization process. Bitterns are often dumped directly into seawater or areas near salt ponds by salt farmers because they are considered unusable. Bittern has a lot of mineral content, namely magnesium chloride ($MgCl_2$), magnesium sulfate ($MgSO_4$), potassium chloride (KCl), sodium chloride (NaCl), and other salts (Ramadhani et al., 2021). Magnesium is one of the most abundant elements in bittern at 8%w/v, so it can be used as a raw material for producing fertilizers (Sanghavi et al., 2020). Based on the Center for Statistics and Information of the Ministry of Maritime Affairs and Fisheries (2018), the total production of salt for pond and non-pond types in Aceh is more than 11 thousand tons, so that can be used as a source of magnesium for struvite as a slow release fertilizer.

Previous studies have explored different raw materials as sources of magnesium, ammonium, and phosphate ions for struvite production. Jagtap and Treavor (2018) varied the sources of ammonium and phosphate, namely urine and without metabolites, as well as human urine. Sources of magnesium were also varied using commercial chemicals, namely $MgCl_2.6H_2O$, $MgCO_3$, and MgO. Edahwati et al. (2020a) used bittern as a source of magnesium and commercial chemicals, namely NH_4OH and H_3PO_4. In addition, Krishnamoorthy et al. (2021) used cow urine and human urine as a source of ammonium, phosphate, and magnesium using a commercial chemical, $MgCl_2.6H_2O$.

The findings from the previous research indicate that the use of waste as a raw material source for struvite production has not been fully optimized. Commercial chemicals continue to serve as sources of magnesium, ammonium, and phosphate. This study aimed to reduce dependence on commercial chemicals by optimizing cow urine waste as a source of ammonium and phosphate, and bittern as a source of magnesium in struvite production. This research examined the storage methods of cow urine and their impact on the struvite crystallization process, a previously unexamined aspect. The use of a cow urine and bittern mixture in struvite production is inadequately addressed in current literature.

This research employed precipitation techniques to facilitate the crystallization of struvite. The method involved precipitating magnesium, ammonium, and phosphate ions from solutions of cow urine and bittern. Cow urine and bittern were also characterized as struvite synthesis materials. The storage time of cow urine and the molar ratio of bittern to cow urine were investigated. The potential of cow urine and bittern in Aceh as a slow-release fertilizer was investigated for plant growth with economically viable production.

2. Methodology

2.1. Materials

The materials used in this research were cow urine obtained from the Kutaraja Aqiqah farm in Rumpet Aceh Besar, bittern solution obtained from the people's salt business in Lambada Aceh Besar Village, aquadest, and NaOH 10 N (Merck 106498, MERCK, Indonesia).

2.2. Raw Material Preparation

The first stage of this research was preparing and characterizing the raw material. Cow urine was used as a source of ammonium and phosphate, while bittern was used as a source of magnesium. Cow urine samples were taken directly from the Kutaraja Aqiqah farm in Rumpet Aceh Besar, and bittern samples were taken directly from the salt business of the people of Lambada Aceh Besar village. The raw materials followed in Figure 1.

Cow urine and bittern were characterized using UV-VIS Spectrophotometric (UV-1800, SHIMADZU) to obtain the concentration of ammonium and phosphate in cow urine and Atomic Absorption Spectroscopy (AAS AA-7000, SHIMADZU) to know magnesium content in bittern.

2.3. Synthesis of Struvite

The synthesis of struvite was conducted by varying two main parameters: the molar ratio of cow urine to bittern (1:1, 1:1.5, and 1:2) and the storage duration of cow urine (fresh, 1 week, 2 weeks, 3 weeks, and 4 weeks) at room temperature. The experimental procedure followed the steps outlined in Figure 2.

Figure 1. Bittern, cow urine, and struvite

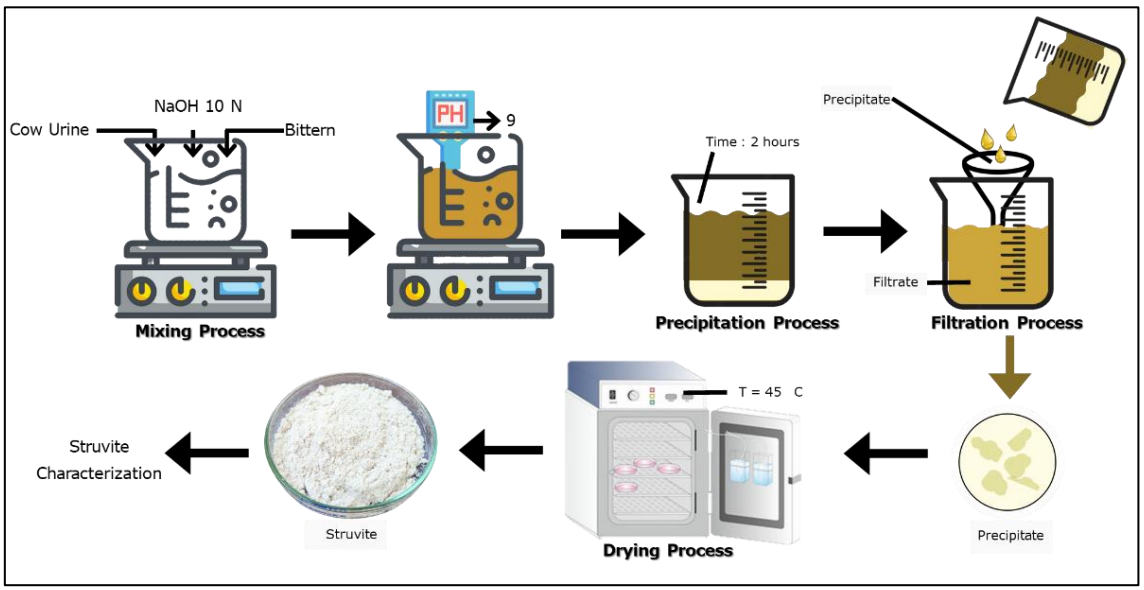

Figure 2. Synthesis of Struvite

Initially, cow urine and bittern were mixed at the specified molar ratios, and the mixture was stirred until a homogeneous solution was achieved. Once homogeneity was reached, 10 N NaOH solution was gradually added to the mixture until the pH of the solution reached 9. The stirring process was maintained at a speed of 158 rpm for 60 minutes, which was controlled using a digital laser tachometer (DT-2234C).

After stirring, the mixture was left undisturbed for 2 hours to allow the formation of a white precipitate. The resulting precipitate was then separated by filtration using Whatman No. 1 filter paper. The filtered precipitate was dried at 105°C in a MEMMERT UN-55 oven for 24 hours to yield the final struvite solid product.

2.4. Characterization of Struvite

The ammonium and phosphate contents in struvite were analyzed using UV-VIS Spectrophotometric instrument (UV-1800, SHIMADZU). The magnesium content in optimum struvite yield was analyzed using X-ray Fluorescence (Lab X-5000, HITACHI). The spectrum of compounds contained in optimum struvite yield was analyzed using Fourier Transform Infrared (IRPrestige-21, SHIMADZU) instrument. Scanning Electron Microscopy (JSM-5410, CAPOVANI) was used to see the morphology of struvite with the optimum yield. Moreover, the type of struvite crystals contained in the optimum struvite yield was found using the X-ray Diffraction (HTK1200N, Optical Systems) instrument.

3. Results and Discussion

3.1. Characteristics of Cow Urine and Bittern

Characterization of cow urine and bittern was essential in determining the suitable composition to obtain struvite at optimum conditions. In this study, the characteristics carried out were pH, ammonia contain, and phosphate contain for cow urine and magnesium contain for bittern. The characteristics of cow urine and bittern can be seen in Table 2.

Table 1 shows that the pH of fresh cow urine was slightly acidic and increases with the length of storage of cow urine, this occurs as a result of hydrolysis of urea (Krishnamoorthy et al., 2021). Urea hydrolysis was the precondition for ammonia recovery from urine. The urease enzyme secreted by bacteria present in urine catalyzes ureolysis leading to formation of ammonium (Tao et al., 2019). Reaction of urea hydrolysis shown in Eq 1.

$H_2NCONH_2 + 3H_2O \rightarrow 2NH_4^+ + HCO_3^- + OH^-$ (Eq 1)

Table 1. Parameter of struvite optimum condition

Parameter	Content (mg/kg)
Phosphate	72.417
Ammonium	0.715
Magnesium	26.000

Prolonged storage results in a reduction in ammonia content due to evaporation. Furthermore, ammonia is oxidised, transforming into nitrite compounds and ultimately nitrate compounds via the nitrification process (Putra et al., 2022). In contrast, phosphate concentration rises with time due to microbial activity decomposing phosphorus, hence increasing phosphate levels (Hastuti et al., 2022).

3.2. Effect of Storage Time of Cow Urine and Molar Ratio on Struvite Content

The molar ratio is very influential on the formation of struvite (Iswarani & Warmadewanthi, 2018). One of the components of struvite was phosphate, sophosphate was used as a parameter to determine the presence of struvite in the sample. The effect of the storage time of cow urine and the molar ratio of the phosphate content of the struvite solid formed can be seen in Figure 3.

Based on Figure 3, it can be seen that the phosphate content obtained decreased along with the addition of the molar ratio between cow urine and bittern. The optimum condition for struvite recovery was achieved using 1 week of urine with a molar ratio of 1:1.0.

The longer of cow urine storage time and the addition of the molar ratio, the lower the phosphate content obtained. This decrease occurs due to the addition of excess magnesium, reducing the purity of the struvite produced (Iswarani & Warmadewanthi, 2018). The parameters of struvite optimum condition can be seen in Table 1.

Furthermore, as depicted in Figure 3 and Table 1, a comparison was drawn between the phosphate content in cow urine and that in struvite. This comparison underscores the struvite formation process from cow urine and bittern, predicated on supersaturation of the solution, wherein concentrations of magnesium ions (Mg^{2+}), ammonium ions

(NH_4^+), and phosphate ions (PO_4^{3-}) exceed their solubility thresholds. Supersaturation ensues from the mixing of solutions with varying concentrations, resulting in combined solutes surpassing stable solubility limits. This condition facilitates the precipitation of these ions, leading to the formation of struvite crystals with markedly elevated phosphate concentrations compared to the phosphate levels initially present in cow urine (Wu et al., 2022).

Table 2. Characteristics of Cow Urine and Bittern

Sample		pH	Ammonia (mg/L)	Phospate (mg/L)	Nitrite (mg/L)	Nitrate (mg/L)	Magnesium (mg/L)
Bittern		7.21	NA	NA	NA	NA	5.5357
Cow Urine	Fresh	6.08	529.00	1.43	NA	NA	NA
	1 week	6.74	10.50	10.87	NA	NA	NA
	2 weeks	7.88	10.44	290.07	345.38	42.28	NA
	3 weeks	8.46	10.32	286.03	246.62	173.52	NA
	4 weeks	8.95	10.40	419.59	224.79	NA	NA

NA: Not tested

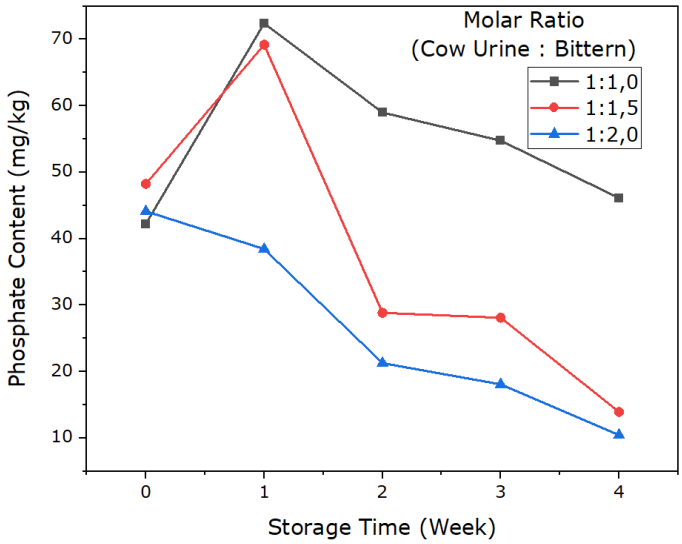

Figure 3. Effect of storage time of cow urine and molar ratio on phosphate content

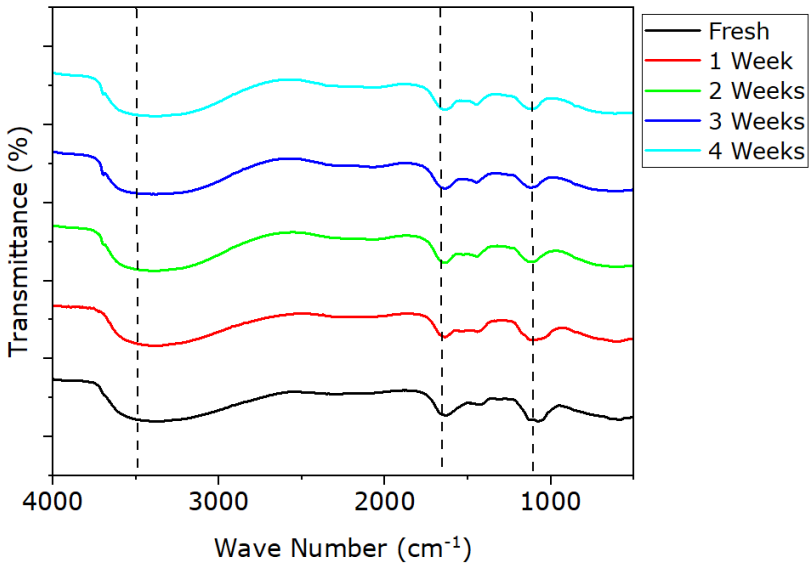

Figure 4. FTIR spectrum of struvite

3.3. Characteristics of Struvite

The characteristics of struvite were observed using FTIR, SEM, and XRD. FTIR was used to examine the spectrum or type of bonding of solid crystals, namely N-H, O-H, and P-O bonds (Atalay et al., 2021). The results of the characteristics of struvite using FTIR on samples of a 1:1 molar ratio of urine and bittern with the storage condition of cow urine (fresh, 1 week, 2 weeks, 3 weeks, and 4 weeks) can be seen in Figure 4. As shown in Figure 4 and Table 3, the transmittance bands within the range of 3550 to 3200 cm^{-1} for struvite correspond to the O-H stretching vibrations of crystallization water. The bands observed around 1650 to 1580 cm^{-1} are indicative of N-H stretching vibrations in the NH_4^+ unit. Additionally, bands appearing in the region of 1195 to 950 cm^{-1} signify the presence of P-O stretching vibrations within the PO_4^{3-} unit (Polat & Perviz, 2019). Thus, these findings confirm that the obtained crystals comprise ammonium, phosphate, and water.

SEM was also used to draw the struvite surface so that the struvite morphology can be enlarged in resolution (Edahwati et al., 2020b). The results of the characteristics of struvite using SEM with a magnification of 500x and 10,000x on the optimum sample, namely the molar ratio of urine and bittern 1:1 with cow urine for 1 week of storage, can be seen in Figure 5.

The SEM results were in the form of surface morphology of the crystals formed as shown in Figure 5, which shows the SEM test results from the front view of the thin layer used at 500x and 10,000x magnification that the crystalline sample structure has a grain size of about 20.7 µm. From the shape obtained, these struvite crystals were grouped into orthorhombic forms characterized by small lumps. Orthorhombic is a crystalline form consisting of three axes of different lengths crossing at an angle of 90° (Adiman et al., 2020). However, some irregular shapes exist because struvite forms impurities or other minerals during the crystallization process (Edahwati et al., 2020b).

Struvite obtained under optimum conditions was also analyzed using XRD to identify powder and solid crystalline phases, which can provide information about diffraction patterns, morphology, and unit cell size in solids (Atalay et al., 2021). The results of the characteristics of struvite using XRD with the help of MATCH software on the optimum sample, namely the ratio of urine and bittern 1:1 with cow urine for 1 week of storage, can be seen in Figure 6. Based on Figure 6, the XRD analysis results show that the crystalline solid profile pattern matches the struvite model needed MATCH Software.

Table 3. Functional Groups of Struvite

Functional Group	Wave Number (cm^{-1})					
	References	Fresh	1 Week	2 Weeks	3 Weeks	4 Weeks
O-H	3550-3200	3469	3573	3491	3506	3520
N-H	1650-1580	1626	1639	1622	1629	1637
P-O	1195-950	1010	1189	1110	1118	1126

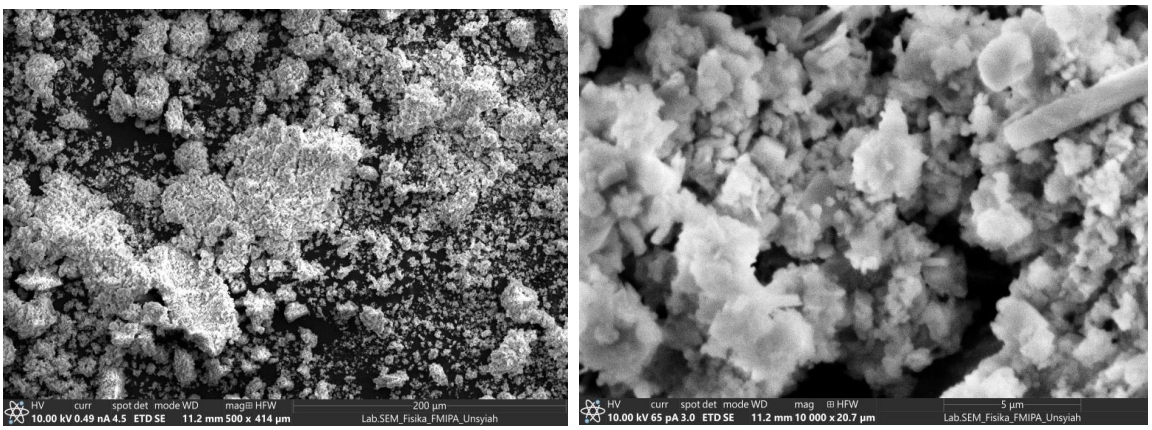

Figure 5. Struvite morphology with 500x and 10,000x magnification.

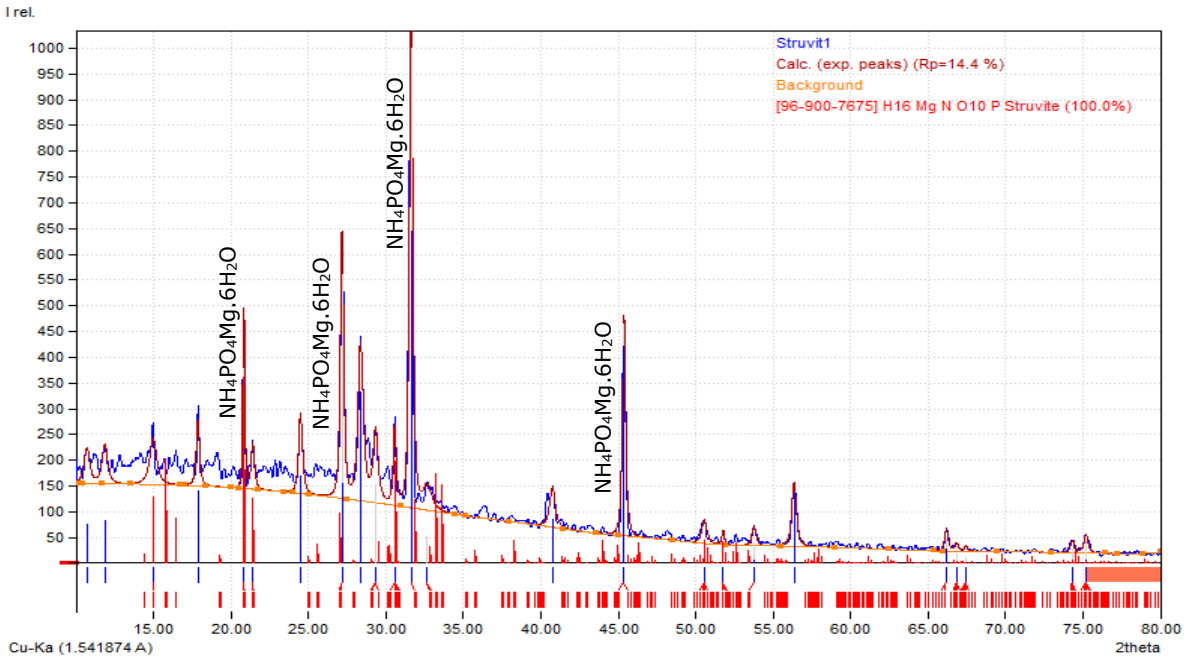

Figure 6. Struvite XRD pattern

Characterization using XRD provided the lattice parameters. In crystallography, lattice parameters a, b, and c represented the dimensions of the unit cell in the crystal lattice, which constitutes the three-dimensional periodic structure of atoms, ions, or molecules within a crystal (Li et al., 2021). Struvite crystallized in orthorhombic form with lattice parameters a = 6.9550 Å, b = 6.1420 Å, and c = 11.2180 Å. However, some peaks in the diffraction pattern did not match, indicating that the tested samples were not completely pure struvite and that other minerals were present during crystallization. The purity level of struvite was also influenced by the condition of the materials used, the type of waste, and other parameters. The initial condition of the waste was brownish-yellow, suggesting the presence of other complex compounds in cow urine and bittern. Consequently, the resulting struvite was impure (Edahwati et al., 2021). Possible impurities present in struvite included Na, K, S, and Cl, which were found in raw material (Le et al., 2024).

3.3. Comparison of the Potential Applications of Synthesized Struvite with Commercial Struvite

The struvite commercial produced by the RUTOM brand (P_2O_5: 28.92% and MgO: 16.43%), available on the market, was used as a reference. Although there are significant compositional differences between the commercial struvite fertilizer from the RUTOM brand (P_2O_5: 28.92% and MgO: 16.43%) and the synthesized struvite produced in this study, these differences do not detract from the potential applicability of the synthesized struvite. The synthesized struvite remains a viable option for agricultural use due to its ability to provide essential nutrients and controlled release properties.

4. Conclusion

Based on the research and data analysis results, it was concluded that the combination of cow urine waste with storage condition variables and bittern could be applied as raw material for the struvite production process because it contains compounds needed for manufacturing struvite. There are struvite functional groups such as P-O, N-H, and water vibrations, and the morphology of struvite is orthorhombic with lattice parameters a = 6.9550 Å, b = 6.1420 Å, and c = 11.2180 Å. In addition, struvite was obtained under optimum conditions for 1-week storage of cow urine and a molar ratio of cow urine to bittern 1:1, namely the ammonium, phosphate, and magnesium content of 72.417; 0.715; and 26.000 mg/kg. This study is expected to advance to plant-based trials to assess the efficacy of its components. It is advisable to enhance the struvite production process to increase efficiency and efficacy. Future study should focus on evaluating the effects of struvite on crop development and yield, exploring appropriate application techniques, and producing comprehensive data on its viability as a sustainable and economical fertilizer option.

Author Contributions

Conceptualisation, Z.F.,; Formal analysis, A.M.A.,; Investigation, T.Z.A.A.,; Project administration, U.Y.A.,; Supervision, -, Writing – original draft, T.M.,; Writing – review and editing, H.

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Data Available Statement

Available on request to the corresponding author.

Conflict of Interest

The author declares that there is no conflict of interest regarding the publication of this manuscript.

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