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The Impact of Urea-Doped Hydroxyapatite Nanomaterials as a Potential Substitute for Traditional Nitrogen and Phosphorus Fertilizers in Acidic Cultivation Environments

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Abstract

The utilization of nutrients in intensive farming is found to be below 50% for macronutrients. The implementation of this feature leads to economic and ecological impacts that are not sustainable. Nanofertilizers have considerable potential as a notable manifestation of nanotechnology within agricultural practices. To utilize nano fertilizers effectively and without risk, it is necessary to understand the specific impacts of nanoproducts on plant metabolism and the subsequent effects on carrier release kinetics and nutrition retention. This study focuses on synthesizing urea-doped hydroxyapatite nanomaterials (UD-HAN) through the sol-gel method. Three different formulations were employed, and the resulting materials were subjected to characterization using Field Emission Scanning Electron Microscope (FESEM) and elemental analysis. To examine the potential fertilizing properties of nanoparticles, UD-HAN was employed across several culture mediums, encompassing alkaline soil, acidic soil, and cocopeat. The study conducted by UD-HAN showed a considerable capacity to serve as a fertilizer in environments with acidic conditions. This work also investigated the nutritional access to synthesized UD-HAN at suggested (UD-HAN-S) and quasi-suggested (UD-HAN-QS) dosages with the suggested dosage of commercial Bulk Urea Fertilizer (BUF) and a Control Treatment (CT) in Acidic Cultivation Environments (ACE). Crop growth characteristics have been evaluated, including plant height, biomass, and production. This suggests that the utilization of UD-HAN has the potential to achieve a yield comparable to that of commercially available fertilizers.

Keywords: *Urea-doped hydroxyapatite nanomaterials, Nitrogen, Phosphorus, Acidic Cultivation Environments, crop yield, Nanofertilizers.*

Introduction

The prevailing agricultural paradigm is founded upon gradually enlarging cultivable territories and heightened utilization of power, fertilizers, pesticides, and water [1]. Nutrient Use Efficiency (NUE) refers to the correlation between the quantity of nutrients plants absorb and the subsequent biomass generation. The NUE for macronutrients in agriculture is often below 50% due to the influence of soil physicochemical features and the specific attributes of commonly employed fertilizers. Due to this factor, the extensive cultivation of crops incurs economic and ecological costs that are not viable in the long term [2]. Nanotechnologies enable the precise regulation and management of materials at the atomic and molecular levels. The unique physicochemical characteristics shown by nanomaterials,

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such as their catalytic reactivity, large surface area, size, and structure, hold promise for addressing challenges in primary farming and enhancing agricultural output [3].

Using nanotechnology inside plant production systems has been designated as "phyto-nanotechnology." Agro fertilization is an area of phyto-nanotechnology that shows significant potential. The term "nano fertilizer" describes a structure with dimensions ranging from 1 to 100 nm, designed to supply macro/micronutrients to crops. Furthermore, it is imperative to broaden the scope of this word to encompass the incorporation of bulk materials in conjunction with nanoscale structures to fabricate novel products [4]. Traditional fertilizers have suboptimal nitrogen absorption efficiency and can contribute to substantial environmental losses. Hence, a crucial aspect of contemporary crop fertilization is mitigating nutrient losses and ensuring optimal synchronization between fertilizer supply and crop absorption. Nevertheless, the potential benefits of nano fertilizers have yet to be empirically shown in real-world agricultural settings [5].

Concerning macronutrients in plants, investigations have been conducted on HAN [$Ca_{10}(PO_4)6(OH)_2$] to examine their possible application as a carrier for nitrogen (N) or as a phosphorus (P) fertilizer. One notable benefit of utilizing HAN compared to other nanomaterials is its well-established reputation for possessing inherent biological compatibility and sustainability since it is a fundamental constituent of human bones and teeth [6]. Therefore, while evaluating the potential use of nanoparticles in open-field applications, it is important to note that the safe biological characteristics of HAN should not give rise to any apprehension regarding human and environmental well-being.

In recent times, researchers have focused significantly on the development of composite nanostructures by combining many components, resulting in the synthesis of a singular nano-system. This approach is precious due to the colloidal nature of the resulting structure, which imparts improved multifunctional capabilities. The capacity to synthesize diverse composite nanomaterials relies entirely on the individual constituents' physiochemical characteristics and the exterior and internal interfacial qualities. Nanocomposite (NC) materials find extensive use in several domains, such as high-energy batteries, inexpensive sensors, conductive paper for extensible batteries, and the facilitation of bone repair [7]. The manipulation of composite materials by adding a greater quantity of constituent elements can result in modifying and enhancing their inherent qualities. The biological characteristics exhibit considerable variability based on the specific magnetic nanoparticles and composites/core shells under consideration.

Agriculture, a fundamental aspect of worldwide food security, has persistent obstacles in maximizing crop productivity while mitigating the adverse ecological consequences associated with traditional agricultural methods. The overutilization of conventional nitrogen and phosphate fertilizers within acidic agriculture settings leads to soil deterioration and groundwater contamination and contributes to greenhouse gas release. In this context, applying cutting-edge nanomaterials as substitutes for conventional fertilizers presents a highly encouraging resolution. This study examines the possible implications of utilizing UD-HANs as an environmentally sustainable alternative to N and P fertilizers in acidic soils. It highlights the significance of UD-HANs in improving agricultural yield while reducing negative environmental consequences.

Literature Survey

This literature review thoroughly investigates the potential implications of incorporating UD-HAN as an environmentally friendly alternative to traditional nitrogen and phosphorus fertilizers in acidic soil conditions. Through a comprehensive analysis of the current body of scholarly literature, our objective is to provide a thorough understanding of the various dimensions of this technology. This examination will encompass its synthesis, utilization, and potential advantages in augmenting agricultural output and addressing ecological concerns.

Tang and Fei (2021) propose making hydroxyapatite nanoparticles from refractory calcium phosphate for phosphorus fertilizer. Implementation involves synthesizing and formulating nanoparticles and using them to deliver plant nutrients. Nanoparticle characterization, nutrient delivery, and plant development are the output results. Nanoparticles regulate phosphorus discharge, reduce environmental impact, and improve nutrient use. This approach has drawbacks, including nanoparticle manufacturing costs and long-term soil health effects.

Maghsoodi et al. (2020) discuss the dilemma of using hydroxyapatite nanoparticles as phosphorus fertilizer. Nanoparticles' effects on plants are assessed using the suggested method. Hydroxyapatite nanoparticles' effects on plant growth and nutrient absorption are tested during implementation. The results reveal the pros and cons of nanoparticles in agriculture. This approach may improve plant well-being, nutrient discharge, and phosphorus runoff, but there may be concerns about nanoparticle toxicity and environmental risks.

In their study, Xiong et al. (2018) suggested customizing hydroxyapatite nanoparticles to improve soil phosphorus fertilization. The technology manipulates hydroxyapatite nanoparticles to improve nutrient transport. Implementation involves creating and using customized nanoparticles in soil. Results include altered nanoparticle characterization, nutrient discharge efficacy, and soil and plant well-being. Benefits of nanoparticles include improved nutritional accessibility and reduced environmental impact. However, nanoparticle manipulation can be complicated and require advanced modification methods.

Avşar (2022) presents a new method for assessing nanohydroxyapatite as an agricultural phosphorus fertilizer substitute. The method involves testing nanohydroxyapatite's nutrient delivery efficacy. The implementation process involves experiments to compare the implementation to traditional fertilizers. The output values show nanohydroxyapatite's phosphorus fertilizer efficacy. This phenomenon may have environmental and nutrient efficiency benefits. Conversely, complete analyses and long-term research may be drawbacks.

Marchiol et al. (2019) examined how hydroxyapatite nanoparticles affect tomato germination and metabolism. Nanoparticles are applied to tomato seeds and plants and then observed for growth and biological reactions. The output values include germination rates, plant metabolism, and crop growth effects. The benefits of using nanoparticles in plant systems include a better understanding of their complex dynamics. One drawback is the need for more research to understand nanoparticles' long-term effects and field use fully.

Noruzi et al. (2023) examine whether hydroxyapatite nanoparticles can replace phosphorus fertilizers in acidic growth conditions. The method involves acidic experiments to assess nutrient release and plant reaction. The output values show hydroxyapatite nanoparticle effectiveness in acidic soil. This method may increase phosphorus availability in acidic soils but may not work for all soil types and pH ranges.

Pohshna and Mailapalli (2021) developed urea-doped hydroxyapatite nanoparticles for rice fertilization. This study synthesizes nanomaterials and uses them to boost rice growth. The output values show how well-designed nanoparticles work as dual-nutrient fertilizers. Using nanoparticles in agriculture improves nutrient utilization and crop yield. This approach has several drawbacks, including the complicated nanoparticle synthesis process and environmental impacts.

The utilization of UD-HAN holds promise for transforming the field of agriculture, as it presents a sustainable alternative to conventional N and P fertilizers in acidic cultivation conditions. The capacity of these innovations to augment nutrient absorption, enhance crop yield, and mitigate ecological damage establishes their significance in advancing sustainable and environmentally conscious agricultural methodologies. To fully realize the potential of UHANs, conducting additional research, engaging in field testing, and performing ecological assessments is imperative. These measures are crucial for achieving the dual objectives of increasing food production and preserving the ecosystem.

Materials and Methods

The synthesis of HAN and UD-HAN was conducted in the Nonpoint Source Pollution Laboratory using the sol-gel method. Fig. 1 depicts the flowchart illustrating the HAN synthesis procedure and urea's subsequent loading to synthesize UD-HAN. The synthesis of HAN involved the utilization of reagents of analytical purity grade obtained from Merck/Sigma Aldrich. These reagents included calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$), tri-ethanol amine, and ammonium dihydrogen phosphate ($NH_4H_2PO_4$). The chemical reaction is depicted in Equation 1.

$$10Ca(NO_3)_2.4H_2O + NH_4H_2PO_4 + NH_4^+ + OH^- \dashrightarrow Ca_{10}(PO_4)_6(OH)_2 + NH_4^+ + NO_3^- + H_2O \quad (1)$$

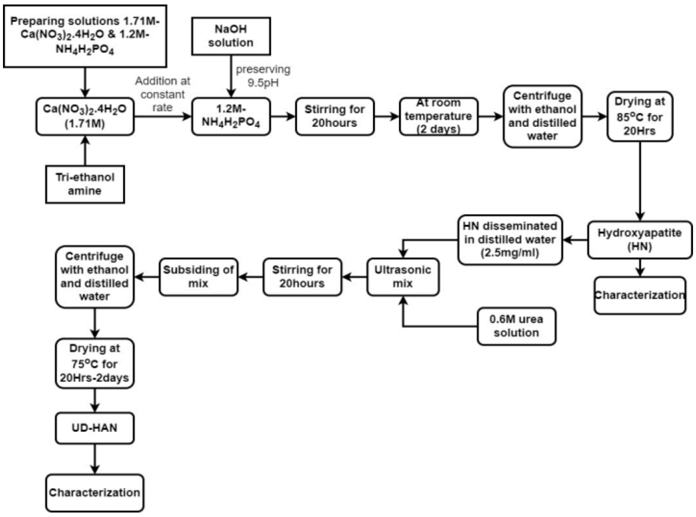


Figure 1: Synthesis of HAN and UD-HAN using sol-gel method

The water utilized in the synthesis procedures underwent a process of distillation and deionization. The experimental approach involves gradually adding a 1.71 M solution of $Ca(NO_3)_2 \cdot 4H_2O$ and ethanol to a 1.2 M solution of $NH_4H_2PO_4$ at a steady rate. Throughout the process, the pH is maintained at 9.5 by adding ammonia. The mixture was stirred firmly under normal circumstances, and the resultant solution was allowed to undergo aging for two days at room temperature. The final solution underwent centrifugation with ethanol and deionized double distilled water to eliminate ammonium and nitrate ions. The filtrate was dried at a temperature of 85 °C for 20 hours using a hot-air oven. This resulted in the production of white powdered HAN, which was subsequently analyzed to ascertain its properties. The urea loading was conducted according to the procedure outlined by Pradhan et al. [15], using a urea-to-HAN ratio of 4:1 by weight. A solution of urea at saturation (0.6 M) was introduced into a solution of HAN dissipated in distilled water (2.5 mg/mL) using ultrasonic mixing (35 kHz for 50 minutes) and subsequently stirred for 20 hours. After 20 hours, the mixture was permitted to undergo sedimentation. The surplus liquid was subsequently separated by decantation, and the resulting product was subjected to centrifugation using distilled water and ethanol to eliminate excess urea. The product was then subjected to a drying process at a temperature of 75 °C for a period ranging from 20 hours to 2 days. The dried samples were subsequently dispatched for characterization.

Field Column Investigations

Field columns were built during the Rabi season (March to June 5, 2021), and the rice cultivar IR-36 tests were carried out. The research site experiences an average of 155.8 cm of annual rainfall, with minimum and maximum daily highs and lows of 11 and 41 °C, respectively, and a humidity range of 15 to 98%. Wind speeds were 3.12 km/h, and sun radiation was 198.1 W/m2 on average.

Stacking of Columns in a Field

13 PVC columns, each measuring 31.2 cm in diameter and 38 cm in length, were used in the trials, which were conducted in an open area (Fig. 2). Leaching to a leachate collection (500 mL bottle) was permitted through a drain hole in the center of the closed column's bottom. The columns were positioned on a platform to allow for easy draining into the collecting bottles.

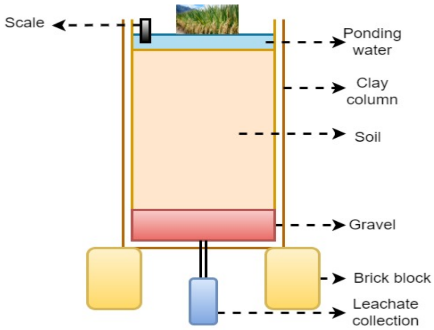


Figure 2: Schematic depiction of a soil column

PVC mesh was placed at the bottom of each column to prevent soil loss. The columns were then filled with pea gravel, reaching a depth of 2.5 cm from the bottom. On top of the pea gravel, a layer of coarse sand measuring 1 cm in depth was added. Subsequently, the columns were filled with puddled soil to a depth of 26 cm, utilizing field soil obtained from the uppermost 26 cm layer of the adjacent paddy fields. The columns remained undisturbed for a designated period to facilitate the correct soil settling. A soil-free zone of 13 cm was maintained at the top of each column to ease water ponding and fertilizer delivery. The soil columns were first saturated with water for 72 hours before the initiation of the tests. Subsequently, the columns were allowed to drain, ensuring uniformity in the beginning conditions of bulk density, porosity, and soil nutrient content throughout all columns.

Treatments

The study examined four levels of nitrogen treatment: a CT, BUF, UD-HAN at the recommended nitrogen dose (UD-HAN-S), and UD-HAN at a quasi-dose of nitrogen (UD-HAN-QS). The experiment was conducted with three replications for each treatment. The UD-HAN was subjected to sonication using distilled water for 1.5 hours, after which it was combined with the ponding water from the column. The prescribed nitrogen dosage was set at 125 kilograms of nitrogen per hectare for all BUF and UD-HAN-S treatments. Conversely, the UD-HAN-QS treatment was assigned a lower dosage of 65 kilograms per hectare. In the BUF treatment, the phosphorus (P) fertilizer dosage was administered using single superphosphate (SSP) at a rate of 65 kg/ha.

Conversely, in the UD-HAN treatments, the quantity of P supplied corresponded to the nitrogen (N) application levels, resulting in 355 KgP/ha in UD-HAN-QS and 185 Kg-P/ha in UD-HAN-QS. The potassium (K) application rate was consistent at 55 Kg K/ha across all fertilizer treatments. Four seedlings, each aged 35 days, were put into every field column. The application of fertilizer treatments occurred on four occasions throughout the experiment. Initially, the basal dose was administered, followed by subsequent applications at intervals of 25 days, a commonly observed duration for a development stage. The magnitude of water accumulation maintained inside the columns exhibited a 1 to 6 cm range throughout the growth phase. The columns were subjected to irrigation until the water depth reached 6 cm, starting from an initial depth of 1 cm.

Soil Properties and Culture Media

Wheat was chosen as the experimental plant species to examine the fertilizing characteristics of HAN. This study aimed to evaluate growth medium, namely a combination of peat moss and perlite (50:50 v/v), cocopeat, acidic soil, and alkaline soil. The soil sample used in this study was obtained from Gilan province in Iran and characterized as a sandy loam soil with a composition of 53% sand, 25% silt, and 22% clay. A sample of alkaline soil comprising sand (33.2%), mud (29.6%), and clay (37.2%) was collected from a nearby field.

Table 1 displays the physico-chemical parameters of the soils. It is worth noting that the soil pH measurements reveal that the acidic soil possesses a pH value of 5.5. In contrast, the alkaline soil exhibits a notably higher pH of 7.7, thus highlighting the discernible differences in their respective degrees of acidity. The nutritional composition analysis reveals that the acidic soil has a nitrogen (N) concentration of 0.24%, notably larger than the alkaline soil's nitrogen content of 0.12%. This discrepancy implies that the acidic soil potentially offers a bigger abundance of nitrogen for plant uptake. On the other hand, it can be observed that the alkaline soil exhibits elevated levels of phosphorus (P) and potassium (K), measuring at 3.8 ppm and 69 ppm, respectively, in contrast to the acidic soil, which displays lower concentrations of 2.6 ppm for phosphorus and 154 ppm for potassium. The observed discrepancies in soil characteristics emphasize the significance of pH levels and nutrient composition in assessing the appropriateness of soil for various agricultural applications, hence emphasizing the necessity for customized soil management strategies.

Table 1: Physical and chemical attributes of acidic and alkaline soils

Properties	Acidic soil	Alkaline soil
Soil pH	5.5	7.7
N (%)	0.24	0.12
C (%)	0.97	0.38
P (ppm)	2.6	3.8
K (ppm)	154	69

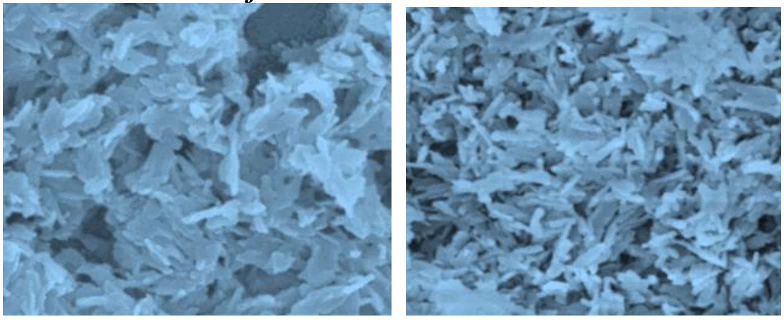
Samples were collected from acidic and alkaline soils at 0-35 cm depth. The collected samples were subsequently air-dried, sieved, and utilized in greenhouse studies. Before planting, the culture media was supplemented with nano-fertilizers and Triple superphosphate (TSP). Before application, none of the nanofertilizers underwent sonication. Before planting, the soils were supplemented with N and K fertilizers at 120 mg/kg and 85 mg/kg. The P treatments were administered at 0 mg/kg (control) or 100 mg/kg.

Results and Discussion

Data Collection

The precipitation data were gathered from the meteorological station situated at the research facility. The ponding water level and leachate volume were monitored daily at 10:00 AM. Water samples were obtained from the stagnant water in the pond and the point of drainage before the fertilizer application. Subsequently, further samples were collected at intervals of 4, 8, 12, and 24 hours and daily throughout the initial week. In the second week, samples were taken every two days, followed by a frequency of every three days in the third week. From the fourth week onwards, one sample was collected each week. Plant height were assessed at regular intervals of 21-24 days. The grain production and biomass of the rice crop were measured by harvesting from each column at the maturity stage, namely 88 Days After Application (DAA). The soil samples, taken from the surface and depths of 18 cm and 30 cm, together with plant samples, were gathered from the columns subsequent to crop harvest to assess nutrient content. The data acquired in the field throughout the 0-55 DAA period were included in the analysis. This decision was made due to column clogging in the later phases of the experiment, namely after 55 DAA. Following the crop harvest, the soil was extracted from the columns, revealing the accumulation of smaller particles at the drain point. This observation suggests the potential for clogging to occur.

Characterization of HAN and UD-HAN



(a)HAN (b) UD-HAN

Figure 3: Characterization using FESEM

The surface morphologies of both HAN and UD-HAN were examined using a Field Emission Scanning Electron Microscope (FESEM). The elemental compositions of HAN and UD-HAN were determined through Energy-Dispersive spectrophotometric (EDX) analysis, as illustrated in Fig. 4. The determination of the shape and structure of HAN and UD-HAN was conducted using a FESEM, as shown in Fig. 3. Fig. 3a displays the FESEM images of the HAN material, revealing a rod-shaped structure. However, it is worth noting that the rods appear clustered due to concentration. The presence of a rod-shaped structure resembling HAN in UD-HAN, as depicted in Fig. 3b, suggests that the morphology of HAN remains unaffected by doping. Additionally, the size of the rod-shaped structure was found to be smaller than 100 nm, which aligns with the findings reported in [15].

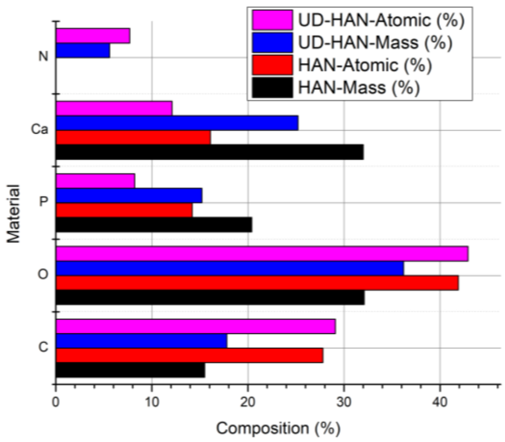
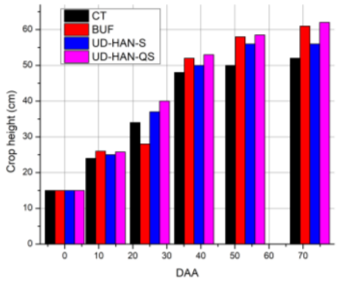
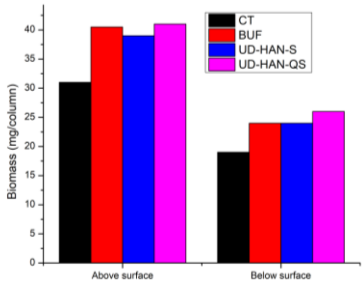


Figure 4: Elemental composition (in %) of HAN and UD-HAN

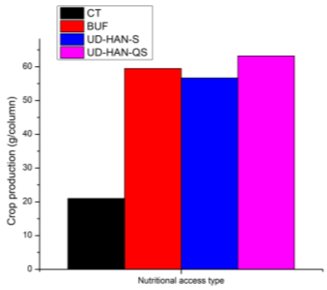
Fig. 4 displays the mass and atomic percentages of the elemental composition of HAN and UD-HAN. The data presented in this study demonstrates notable disparities in the elemental composition of the two materials. Regarding HAN, the primary constituents consist of carbon (C), oxygen (O), phosphorus (P), and calcium (Ca), with oxygen being the most abundant element in terms of both mass and atomic percentage—nevertheless, the implementation of urea doping in UD-HAN results in significant alterations. The carbon and oxygen content of the material experiences a moderate increase. In contrast, the nitrogen (N) content becomes a significant constituent, accounting for 5.6% of the material's mass and 7.7% of its atomic composition. The observed change in elemental composition highlights the impact of urea doping on the chemical structure of hydroxyapatite nanomaterials, potentially altering their properties and applicability for diverse uses.

Crop Growth Results in ACE





(a)Crop height (b) Biomass



(c) Crop production

Figure 5: Crop Growth results in ACE for fertilizer treatments: CT, BUF, UD-HAN-S, and UD-HAN-QS

Fig. 5a shows how fertilizer treatments affect crop height over time. Beginning on DAA 0, all treatments begin with a consistent height measurement of 15 cm. However, as the DAA progresses, crop height disparities become noticeable. UD-HAN-QS consistently outperforms other treatments. It always has the highest crop height values across DAA, peaking at 62 cm at DAA 72. This suggests that UD-HAN-QS has a greater and longer-lasting impact on crop growth than other treatments. However, the CT treatment has the lowest growth rate, highlighting the need for fertilizer amendments to increase crop height over time.

Fig. 5b shows how fertilizers affect biomass accumulation, distinguishing between above- and below-surface components. UD-HAN-QS consistently produces the most biomass, up to 41 mg/column. BUF treatment increases biomass to 40.5 mg per column. The CT treatment has the lowest above-surface biomass, suggesting limited growth without fertilizer. Below-surface phenomena show similar patterns, with UD-HAN-QS and BUF outperforming the others. This study found that UD-HAN-QS and BUF can increase biomass above and below the surface.

Fig. 5c shows crop production output. UD-HAN-QS produces the most at 63.2 g/column. BUF delivers 59.5 grams per column. At 21 g/column, the CT treatment has the least crop output. Results emphasize the importance of fertilization for crop yield and nutritional availability. The results show that UD-HAN-QS and BUF fertilizer techniques provide enough nutrients to plants, increasing crop yield compared to the control group. These findings highlight the importance of choosing the right fertilizer strategy to maximize crop growth in agriculture.

Conclusion

The primary objective of this work is to examine the production of UD-HAN utilizing the sol-gel technique. Three distinct formulations were utilized, and the resultant materials underwent characterization FESEM and elemental analysis. The investigation of nanoparticles' fertilizing capabilities involved using UD-HAN in various culture media, including alkaline soil, acidic soil, and cocopeat. The UD-HAN has demonstrated a significant potential for utilization as a fertilizer in areas characterized by acidic conditions. The carbon and oxygen content of the UD-HAN experiences a moderate increase. The nitrogen (N) content becomes a significant constituent, accounting for 5.6% of the material's mass and 7.7% of its atomic composition. UD-HAN-QS produces the most at 63.2 g/column. BUF produces 59.5 grams per column. At 21 g/column, the CT treatment produces the least crop output. The results emphasize the importance of fertilization for crop yield and nutritional availability.

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