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Preparation, Characterization and Nitrogen Release Behaviour from Urea Ammonium Nitrate Loaded Poly (Acrylic Acid-co-Acrylamide)/ Bentonite Based Nanoclay Polymer Composites in Soil

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ABSTRACT

This laboratory study was conducted during October, 2023 to December, 2023 at Indian Agricultural Research Institute, New Delhi and focused on the synthesis, characterization, and evaluation of poly (acrylic acid)-co-acrylamide / bentonite based nanoclay-polymer composites (NCPCs) for their potential application as slow-release nitrogen fertilizers. NCPCs were synthesized by co-polymerization reaction between partially neutralized aqueous acrylic acid solution with acrylamide and bentonite clay (in varied amount; 8, 12 and 16% by weight of acrylic acid), in presence of crosslinker and free radical reaction initiator. The composites were characterized using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) techniques for physical properties. The water absorption and retention studies revealed high swelling capacities of the NCPCs, enabling gradual water and nitrogen release. Nitrogen release studies revealed that NCPCs loaded with urea ammonium nitrate (UAN) in soil showed a slower (40–60% less total N release over 28 days) and more sustained release compared to conventional UAN fertilizer. NCPCs regulated nitrogen availability by releasing ammonium ($NH4^+$) and NO_3 at a slower rate during the incubation period, but the effect was more visible upto 15 days. Further N release from UAN-loaded studied using zero-order, first-order and Higuchi's kinetic reaction model; results highlighted that N release rate followed first-order kinetic reaction evident from higher R^2 (0.93) over others. In conclusion, NCPCs can sustain N availability from UAN in soil for longer duration and offers its suitability for agricultural application for better nutrient use efficiency over conventional UAN fertilizer.

KEYWORDS: Urea ammonium nitrate, slow-release fertilizer, nanoclay-polymer composites (NCPC)

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1. INTRODUCTION

Fertilizers are indispensable for enhancing crop production and maintaining productivity, with nitrogenous fertilizers like urea being widely used due to their low cost and high nitrogen concentration. However, significant environmental losses of applied nitrogen occur through processes such as surface runoff, leaching, and gaseous emissions, resulting in nitrogen utilization efficiencies of only 30–40% depending on soil conditions (Congreves et al., 2023). This inefficiency leads to substantial economic losses and environmental concerns, including groundwater nitrate pollution, eutrophication, and greenhouse gas emissions (Craswell, 2021). To mitigate these issues, advancements such as large granular fertilizers, polymer coatings (e.g., sulfur-coated urea, IBDU), and nitrification inhibitors (e.g., N-serve, Nitrapyrin, DCD) have been developed (Abalos et al., 2014; Alonso-Ayuso et al., 2016 and Rose et al., 2018).

In recent years, the development of polymer composites based on acrylic acid and acrylamide has gained attention due to their superior water absorption and nutrient-holding capacity associated with lower nutrient diffusion rate from nutrient loaded polymer matrix (Liang et al., 2007; Singh et al., 2011). Superabsorbent are three dimensionally cross-linked hydrophilic polymers that are insoluble, hydrogel forming and capable of absorbing large amount of water Loading of nutrient solutions into superabsorbent polymer networks and their application for crop production reported to increase nutrient uptake and nutrient use efficiency. These superabsorbent hydrophilic polymers, though effective in improving nutrient use efficiency, suffer from poor biodegradability and high synthesis costs if solely synthesized using pure chemicals. Incorporating inorganic clays like montmorillonite, attapulgite, kaolinite, into these polymer networks improves their physical stability and degradation rates in soil (Lee and Yang, 2004; Li et al., 2004 and Liang and Liu, 2007).

Pot culture and field studies had shown that nanoclay polymer composites (NCPCs) loaded with urea exhibit slower nitrogen release, enhancing nitrogen use efficiency and crop yields in crops like pearl millet, maize and wheat respectively (Sarkar et al., 2015; Kumar et al., 2024). But in all those studies showing slow-release behavior of N loaded nanoclay polymer composites used urea as their N source. Urea by itself contains N in organic form, release N in plant available inorganic form after transformation in soil; which again takes some time (2–7 days generally) depending on soil conditions (Sankhayan and Shukla, 1976; Singh and Bajwa, 1986). Thus, NCPC loaded with urea by virtue of its organic N source releases N at a slower rate; which raises question about actual potential of NCPCs to be used as slow-release fertilizer form. Therefore, it will be more logical to use a N source containing readily available form; urea ammonium nitrate (UAN), containing 50% inorganic nitrogen (NH_4NO_3) and 50% organic nitrogen (urea) perfectly fits in this role. A comprehensive study on N release pattern form UAN loaded NCPCs will actually highlights the role of NCPCs as slow-release fertilizer in soil.

In this background we synthesized NCPCs using acrylic acid + acryl amide+inorganic clay and loaded them with UAN and observed N release pattern from them. We prepared NCPCs using three clay concentrations i.e., 8, 12 and 16% by weight (of acrylic acid). Objectives of this study are (i) how slow or fast N released from UAN loaded NCPC as compared UAN in soil, (ii) to study the effect of the inclusion of inorganic clays in the poly[(acrylic acid)-co-acrylamide] NCPC matrix on the N release behaviour of these polymer composites.

2. MATERIALS AND METHODS

2.1. *Experimental location and time*

This laboratory experiment was conducted in Division of Soil Science and Agricultural Chemistry, Indian Agricultural Research Institute, New Delhi, India (28.08°N and 77.12°E) during October 2023 to December 2023.

2.2. *Preparation of nano clay polymer composites (NCPC)*

The NCPCs were synthesized by copolymerization reaction of partially neutralized acrylic acid (AA) and acrylamide (AM) as described by Liang and Liu (2007) with some modifications. Briefly 55 ml acrylic acid neutralized with 24 ml ammonia solution in order to achieve 60% neutralization in a four-neck vial linked with nitrogen gas supply. After the neutralization reaction was completed, 11.5 g of acrylamide and required amount of commercial bentonite as (8% and 10% by weight of acrylic acid) were added into the reaction vessel. Then the reaction vessel was then placed in a magnetic stirrer cum heater (450 rpm) and heating started at 80°C. In the next step methylene bisacrylamide (0.5 g) (N,N-MBA) was added in the reaction vial as cross linker (between acrylic acid and acryl amide) and heating initiated in presence of continuous N_2 gas supply. After 30 minutes, ammonium persulphate (APS) (0.8 g) added in the reaction vial as Free radical initiator and heating increased to 110°C until polymerization reaction completed. After polymer formation, final product kept in a petriplate and cooled, oven dried until it lost all moisture.

2.3. *Characterization of NCPCs*

Synthesized NCPCs were characterized by Fourier transform infrared (FTIR) spectrophotometer at 4000 - 450 cm^{-1} wavelength range (24 scans resolution^{-4}, cm^{-1}) (Brooker India Scientific Private Limited, Mumbai, India), X- ray diffraction analysis of the NCPC samples were carried out by X-ray powered diffractometer with a Cu anode (Philip PW 1729) running at 40 kV and 20 mA, with 3- 40° scan angle, scan type-continuous.

2.4. Water absorption and retention capacity

Place 1 g of NCPC samples in a 150 ml beaker, add 100 ml water and allow NCPC molecules to absorb water until they reach swelling equilibrium for 48 hours; and then filter out excess water until free water droplet movement halted.

The following equation was used to determine the equilibrium water absorbency (QH_2O):

Q_{H2O} (g g^{-1})=$(W_2-W_1)/W_1$ (1) ------------------ (Eq-1)

Where, W1 is the weight of NCPC and W2 is the weight of swollen NCPC. Q_{H2O} was expressed as grams of water absorbed g^{-1} of sample.

2.5. Nitrogen release in soil

N release from UAN and UAN loaded NCPCs were evaluated using a laboratory incubation study. Briefly, UAN containing 100 mg N (0.33 ml) was added into 10 ml distilled water and then the UAN mixed solution added into 50 ml beaker containing 1 g dry NCPC products; kept for 72 hrs to obtain proper UAN loaded NCPC product. This UAN loaded NCPCs then placed in a beaker (150 ml) containing 100 g soil in such a way that after addition of 50 g soil, UAN loaded swollen NCPC products added and then remaining 50 g soil added in such a way that NCPC product fully covered with soil. Then required amount of water 30 ml) added in those beakers to maintain moisture level at maximum water holding capacity (based on the fact that irrigation applied after fertilizer application in crop field and cropped plots received irrigation until soil become fully saturated). Mineral N released in soil was analyzed after 3, 7, 14, 21, and 28 days of incubation. For N release from UAN, we took 0.33 ml UAN (containing 100 mg–N) and mixed in 30 ml water and added in the same quantity soil. To eliminate N from native soil, we also took same amount of soil without any UAN loaded NCPCs product in replicated beakers. N released from urea and urea-loaded NCPC products are obtained upon subtraction of N contributed from the soil itself. Destructive sampling was carried out during sample collection for mineral N estimation. Ammonical (NH_4^+) and nitrate (NO_3^-) N released in the soil were extracted with 2M KCl solution (Kinney and Nelson,) and determined by steam distillation method using MgO and devarda alloy.

2.6. Modeling N release kinetics

N release behaviour from UAN loaded NCPC products evaluated through first-order, zero-order and Higuchi kinetic models.

2.6.1. First-order release kinetics

In first-order reaction model assumes that nutrient release rate (change in nutrient concentration over a period of time) depends on the initial concentration. Nutrient release pattern followed the first-order model generally expressed using the below equation:

$C_t=C_0\ (1-e^{-k1t})$ ---------------------------------- (Eq-2)

Where, Ct is the cumulative nutrient released at time t, C0 is the amount of potentially mineralizable nutrient in the fertilizer, and k1 is the first-order rate constant.

2.6.2. Zero-order release kinetics

In zero-order reaction model hypothesized that nutrient release rate will be constant over a period of time and release rate does not depend on initial concentration, can be expressed using the below equation.

$Q_t=Q_0+k_0t$ -------------------------------------- (Eq-3)

Where, Qt is the amount of nutrient released in time t, Q_0 is the initial amount of nutrient in the solution / medium, and k0 is the zero-order release constant. The model was examined by plotting the cumulative nutrient release (Q_t) against time (t). The slope at which the graph is linear can be used to determine the value of the release constant, k_0.

2.6.3. Higuchi release kinetic model

Higuchi model follow the principle of Fick's law diffusion and generally used to assess drug release behaviour. This model based on the assumption that, release of a substance from an insoluble matrix is proportional to square root of time and generally expressed as

$Q=kHt^{1/2}$ --------------------------------------- (Eq-4)

Where, Q can be represented as $Mt/M\infty$. Mt represents the nutrients released at time t, and M represents those released at time infinity. kH is the Higuchi dissolution constant (Higuchi, 1963).

2.7. Statistical analysis

One way Analysis of Variance (ANOVA) was carried out by windows-based SPSS 17 package followed by Tukey's HSD test as post hoc mean separation test. For drawing the N release figures and for model fitting we used windows-based Microsoft Excel 2007.

3. RESULTS AND DISCUSSION

3.1. FTIR spectroscopy

The FTIR spectra of bentonite clay, acrylamide and NCBPC made up with 8, 12 and % clay were shown in Figure 1.

Typical characteristic peak of bentonite appeared at 1032 and 500–700 cm^{-1} wavelength respectively related/ linked with Si-O stretching vibrations of the Si-O-Si

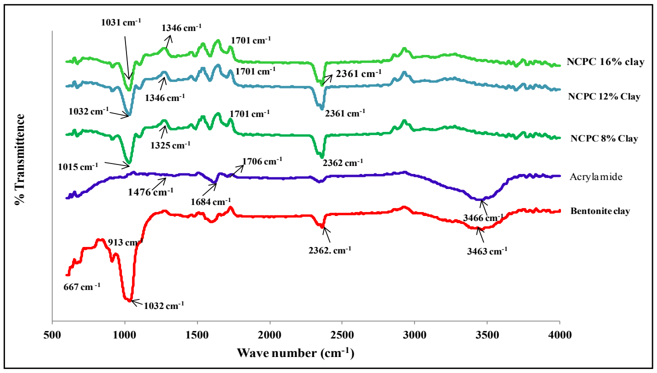

Figure 1: FTIR spectra of bentonite, acrylamide and different nanoclay- polymer composites (8, 12 and 16% clay)

tetrahedra and angular deformation of Si-O-Al bond in the octahedral sheet respectively (Li et al., 2005; Liang and Liu, 2007). Apart from these in bentonite, spectral peak appeared at 3600–3800 cm^{-1} linked with structural hydroxyl (-OH) groups associated with such as Mg-OH, Al-OH or Fe-OH which is typical signature of smectitic or montmorillonitic clay minerals (Sarkar et al., 2013). In acrylamide, characteristic peak appeared at the region of 3200–3600 cm^{-1} due to presence of hydroxyl groups (hydrogen bonded) and weak peak appeared at 1500–1700 cm^{-1} due to presence of carbonyl (C=O) or amide groups ($-NH_2$) (Liang and Liu, 2007). FTIR spectra's of NCPC products (8, 12 and 16% clay) showed weakening or disappearance of the characteristic peaks of bentonite (1032 cm^{-1} and 3300–3600 cm^{-1}) and acrylamide (3300–3600 cm^{-1}) confirmed the formation of new product, where some typical characteristic properties of the base molecules either got missing or suppressed. Apart from this, disappearance of typical spectral peaks of bentonite (1032 cm^{-1}) and acrylamide (3300–3600 cm^{-1}) accompanied with appearance of distinguishable stronger or intensified spectral peaks at 3600–3800, 2400 and 1400–1600 cm^{-1} zones indicates graft polymerization of between clay and poly-acrylamide monomers takes place during the copolymerization reaction and ultimately resulted formation of a new product retaining properties from both clay and acrylamide monomers (Li and Wang, 2005; Li et al., 2005; Zheng et al., 2007). More specifically, small peak seen in bentonite at 2400 cm^{-1} and acrylamide at 1400–1700 cm^{-1} go intensified in the finally synthesized molecules (Mandal et al., 2021).

3.2. XRD analysis

The X-ray diffraction was done to understand the crystal nature, periodicity, and polymerization in newly synthesized products. Bentonite showed characteristic peak at 6.4° diffraction angle (2θ) (d spacing=13.8 A°) confirmed the presence of montmorillonites or smectites in the clay lattice (Figure 2).

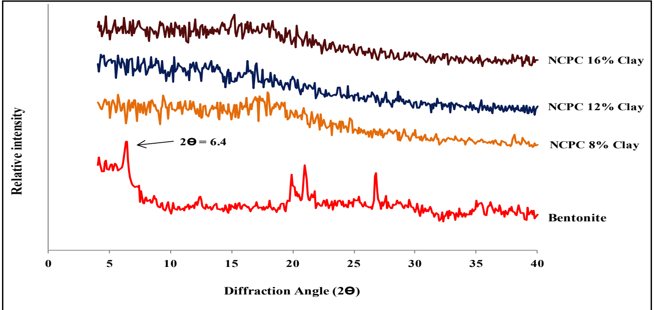

Figure 2: X-ray diffraction pattern of bentonite, acrylamide and different nanoclay- polymer composites (8, 12 and 16% clay)

All NCPC products containing 8, 12 and 16% clay showed similar X-ray diffraction pattern, with no specific peak found in them (Figure 2). The characteristic peak of bentonite clay at (2θ=6.4°) was absent in the case of polymer composites indicative of the complete dispersion or inclusion of bentonite clay in polymer metrics network and the exfoliated nature of polymer composites. The absence of diffraction peaks in the XRD diffractograms also suggests formation of excessive large spacing between two lattice layers in the nano-composite structures due to binding with clay and polymer networks of exfoliated structures. Previously many researchers reported the similar observations (Li and Wang, 2005; Liang and Liu, 2007; Zhang et al., 2007).

3.3. Water absorption and retention capacity of polymers

We observed that with increase in clay content, water absorption capacity of NCPC molecules decreased. The maximum water absorbency was observed in NCPC (8%) (42 g g^{-1}), followed by NCPC (10% clay) (38 g g^{-1}) and NCPC (16) (32 g g^{-1}). Previous researchers reported that similar observations and our findings are aligned with them (Sarkar et al., 2015). Absorbed water released gradually with time if that water soaked swollen NCPCs kept in air (in response moisture level present in the atmospheric air until equilibrium obtained) and takes 12-13 days almost 90% absorbed water got released; after that no further reduction in weight was observed. On the 15th day of incubation, the moisture content of the NCPC-8% clay, NCPC-12% clay, and NCPC-16% clay was 2.12 g g^{-1}, 2.24 g g^{-1}, and 2.31 g g^{-1}, respectively.

3.4. Nitrogen release in soil and release model

There was sharp increase in NH_4 release observed from UAN at 7 days after incubation, followed by rapid decline in NH_4 at 15 days, and progressively decreasing at later incubation period i.e., 21 days and 28 days respectively (Figure 3a). All three NCPCs (8, 12 and 16% clay) showed 37–51% lower NH_4 release compared to conventional UAN especially initial 4th and 7th days after incubation (Figure 1). But as compared to 4th days, sharp increase in NH_4 content observed at 7th days after incubation in all NCPC molecules as observed in conventional UAN incubated soil. At 15 days, NH_4 released from conventional UAN and all UAN loaded NCPCs decreased as observed in 7 days, but still the conventional UAN still maintain 40 - 46% greater NH_4 over UAN loaded NCPCs. At 15 days, NH_4 release from UAN decreased by 49.87% (263 mg kg^{-1}); while 38.76–52.26% reduction in NH_4 content was detected in UAN loaded NCPC's (8, 12 and 16% clay) respectively as compared to 7 days. After 15 days, NH_4 content gradually decreased upto 28 days from both UAN and UAN loaded NCPC's respectively (Figure 3b). All though, UAN loaded NCPCs exhibit lower NO_3 release as compared to conventional UAN, although the gaps between NO_3 was lesser (63–73%) in the initial incubation days (4th and 7th days) and widened (81–86%) after 15 days after incubation. This phenomenon was true for all NCPCs; indicating lower nitrification rate in NCPCs. Overall, UAN loaded NCPCs maintained 45–63% lower total N release over conventional UAN during the incubation period. Clay content did not have any effect in N release (Figure 3a, 3b and 3c).

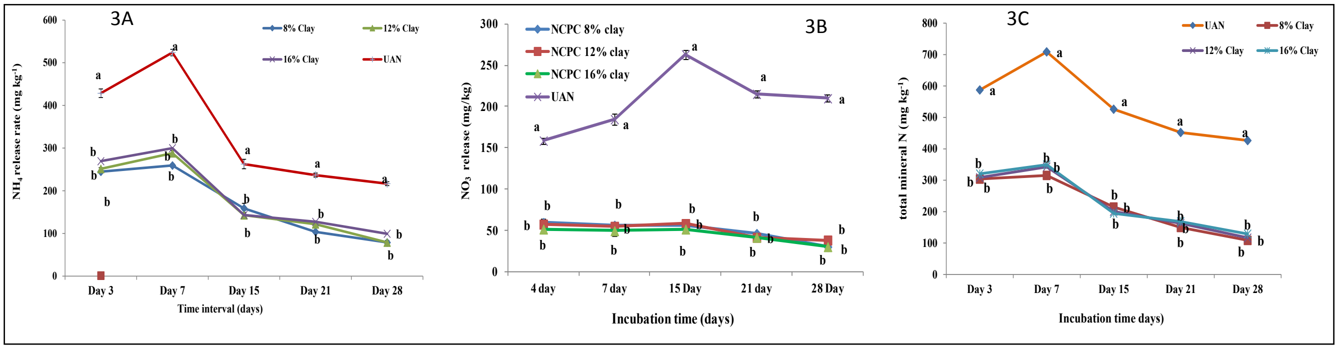

Figure 3: NH_4, NO_3 and total N release behaviour from conventional UAN and UAN loaded NCPC's (8, 12 and 16% clay) at different intervals of incubation study. Tukey's HSD test performed for post-hoc separation of mean

In our study, we observed that NH_4-N release from UAN incubated soil samples far more (over NCBPC loaded UAN incubated soil; the effect was more visible at initial 3 and 7 days. This phenomenon occurs due to chemical composition of UAN, as UAN contains 50% urea and 50% NH_4NO_3; thus, a portion of inorganic N present in as such NH_4 form readily available in the soil, explains higher NH_4 content in the initial 3 days. After 3 days, sudden sharp increase in NH_4 content at the 7 days of incubation related to hydrolysis of urea and followed by rapid release of NH_4 in soil (assuming that, urea hydrolysis takes 3–7 days depending on soil conditions like moisture and temperature) (Dalal, 1974; Prasad, 1976; Dalal). After 7 days, sudden drop in NH_4 content and gradual increase in NO_3 in UAN treated soil may be due to possible transformation NH_4 to NO_3 (nitrification) (Puchades et al., 1984). Unlike conventional UAN, UAN loaded NCPCs resulted slower NH_4 in soil especially in the 3 and 7 days and later maintained a constant release rate in the subsequent incubation period. Because, NCPC product tend to absorb water, >25 (35–42 g g^{-1}) times of their dry weight and release absorbed N (urea) from their pore structure only when soil become gradually dry; moisture suction becomes higher than critical limit and water from polymer structure released in soil in response to water potential gradient. Therefore, from all NCPC products, first NH_4 and urea released in soil with along with water gradually at slower rate coupled with release of polymer absorbed water and subsequently that released urea subjected to hydrolysis and ultimately released NH_4 in the soil system. These mechanisms ultimately lowered the NH_4 release from UAN loaded NCPCs in soil compared to conventional UAN. Further, we observed very low NO_3 content from all NCPCs, indicating lower nitrification rate specially at 21 and 28 day may be linked with moisture shortage or soil drying, may be moisture suction created at this time not exceeds the critical limit to release water from polymer matrix (Breuer et al., 2002; Norton and Ouyang, 2019). In addition to moisture shortage, lower air temperature (<20°C) might have negatively affected nitrification process. We conducted this in the November-December, when night temperature falls below 10°C and during day also temperature hardly raised more than 20°C inside room may be negatively affected nitrification process (Shammas, 1986 and Golden et al., 2011). Previously slower release of N from NCPC loaded N fertilizer products were obtained by Sarkar et al., 2013; Saurbh et al., 2015; Saini et al., 2024. However, total mineral N release decreased with time; this may be linked with gaseous loss of nitrogen in the form of ammonia or lower volume of soil sample taken for mineral estimation instead of full volume soil used in incubation study. Since probability of NH_3 volatilization was low due to lower atmospheric temperature, but we did not study gaseous N loss. Other hand, although we had taken 100 g dry soil for incubation study, but for mineral N estimation we used only 70 g soil for better handling and discarded 30 g soil which closely adhered to the NCPC products; which may lead to lower N recovery specially at later sampling days. Due to presence sufficient moisture, these phenomena did not affect total N recovery in the initial sampling days; but with moisture stress affected at later sampling periods.

Data regarding N release kinetics models (R^2 and reaction constant) were given Table 1. Based on the results obtained from the fitting of different kinetic models, it was observed that first-order release kinetics performed (R^2=0.93) over zero-order (R^2=0.37) and Higuchi reaction kinetics model (R^2=0.35) (Table 1). This indicates that release of N from UAN loaded NCPC products depends on initial concentration of N added into polymer matrix. Our results are in agreement with Wei et al. (2019), reported that N release behaviour from polymer composites prepared from potato starch grafted acrylic acid-acrylamide + maleic hydrazide + halloysite nano tubes follow first-order release kinetics model.

Table 1: Estimates of the Kinetic models parameters for the inorganic N release from UAN, UAN loaded NCPC 8, 12 and 16% clay

Fertilizer product	Nutrient release models along with kinetic parameters							
	Zero order			First order reaction			Higuchi model	
	Q_0	K_0	R^2	C_0	K	R^2	kH	R^2
UAN	837.88	- 20.02	0.79	509.20	- 6.86	0.65		
NCPC 8% clay	625.84	- 22.76	0.56	108.8	- 79.98	0.93	0.240	0.37
NCPC 12% clay	630.10	- 22.28	0.55	226.37	- 6.61	0.93	0.269	0.35
NCPC 16% clay	633.10	- 22.15	0.55	232.45	- 6.62	0.93	0.264	0.35

4. CONCLUSION

NCPC-loaded UAN as a promising option for enhancing nitrogen retention by reducing its release rate. The controlled release, described by a first-order kinetic model, ensured sustained nutrient supply, minimizing N losses and improving efficiency. NCPC-loaded UAN offered possible benefits such as improved crop uptake, reduced environmental impact, and enhanced soil fertility.

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