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RESEARCH ARTICLE

Aqueous release kinetics of nitrogen and potassium from a citric acid–crosslinked lignosulfonate biopolymer matrix

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Abstract

Conventional nitrogen and potassium fertilisers exhibit rapid dissolution in aqueous environments, resulting in low utilisation efficiency and significant nutrient losses. In this study, a citric acid–crosslinked lignosulfonate biopolymer matrix containing nitrogen and potassium was synthesised and evaluated for its controlled nutrient-release properties in aqueous media. The biopolymer fertiliser was evaluated against conventional fertilisers (urea and muriate of potash) under varying pH (4.0, 7.0 and 9.0) and temperature (25 °C and 35 °C) and water conditions (distilled and saline at 2 dS m^{-1}). Under neutral conditions, cumulative nutrient release reached 93.73 % for nitrogen and 83.42 % for potassium at pH 7.0, whereas release declined under acidic and alkaline pH conditions due to changes in polymer swelling and nutrient diffusion dynamics. Temperature significantly influenced nutrient release, with maximum cumulative release observed at 35 °C, reaching 97.32 % for nitrogen and 89.12 % for potassium. In contrast, saline water conditions (2 dS m^{-1}) reduced total nutrient release to 76.31 % nitrogen and 72.53 % potassium by day 30. Conventional fertilisers such as urea and muriate of potash (MOP) released more than 99 % of their nutrients within 3 days. Kinetic modelling revealed that nutrient release from the polymer matrix followed anomalous (non-Fickian) transport and was best described by the Korsmeyer-Peppas model. Furthermore, a maize hydroponic bioassay demonstrated improved seedling performance, with 96.23 % germination, longer root and shoot lengths, higher seedling vigour index (SVI) (1673.91) and increased Soil Plant Analysis Development (SPAD) chlorophyll value (39.80). These findings demonstrate the fertiliser's controlled-release efficiency, environmental safety and potential for precision agriculture.

Keywords: biopolymer fertiliser; controlled release; hydroponic bioassay; nutrient use efficiency; polymer swelling

Introduction

Global fertiliser consumption has risen sharply in response to escalating food demands, which are projected to increase by 59–98 % by 2050 as the global population approaches 9.7 billion (1). In 2022, inorganic fertiliser use exceeded 185 million tonnes, reflecting a 59 % increase since 1980. Notably, nitrogen and potassium fertilisers critical for sustaining crop productivity, increased from 60.6 Mt to 108.1Mt (+78 %) and from 11.2Mt to 16.3Mt (+46 %) respectively (2). Nitrogen fertilisers contribute to 40–60 % of global food production, while potash (K_2O) accounts for over 95 % of agricultural potassium use (3). By 2024, global production of nitrogen and potash fertilisers are projected to reach 199.7 Mt and 73.5Mt, respectively (4). Despite these high application rates, nutrient use efficiency (NUE) remains suboptimal: only 30–40 % of applied nitrogen and 50–60 % of potassium is taken up by crops, with the remainder lost through volatilisation, leaching, runoff, or immobilisation (5). These inefficiencies reduce agronomic effectiveness and contribute to soil degradation, elevated production costs, water pollution and increased greenhouse gas emissions (6).

Controlled release fertilisers (CRFs) offer a promising solution by synchronising nutrient availability with crop uptake while minimising environmental losses (7). The CRFs typically regulate nutrient release through polymeric coatings or matrices that function via diffusion, swelling, or degradation mechanisms (8). Conventional petroleum-derived polymers like polyethylene and polyacrylamide provide controlled release, but suffer from non-biodegradability, ecological persistence and high production costs (9). In contrast, biopolymer-based CRFs derived from lignin, cellulose and starch present a more sustainable alternative due to their biodegradability, cost-effectiveness and environmental compatibility (10, 11). These systems have demonstrated enhanced crop yield (+5.1 %), nutrient uptake (+7.1 %) and reductions in greenhouse gas emissions (-3.6 to -18.6 %) and nutrient losses (-32.6 to –49.1 %), with a market Compound Annual Growth Rate (CAGR) of 6.37 % (12).

Among various biopolymers, lignosulfonate-a sulfonated lignin derivative from the sulphite pulping process-holds considerable potential due to its high cation exchange capacity, functional group density (-COOH, -OH, -SO_3H) and film-forming ability (13). Cross-linking lignosulfonate with citric acid, a non-toxic tricarboxylic acid, enhances its matrix integrity through esterification and ionic interactions, improving mechanical strength, water resistance and swelling behaviour (14, 15). Recent studies report that lignin matrix-based CRFs can reduce ion solubility and delay nutrient release by 20–30 fold compared to uncoated fertilisers (13, 16). Similarly, previous researchers reported a 50 % reduction in NH_4^+ and 46 % in NO_3^- leaching when using lignin-based matrix-bound fertilisers compared to standard formulations (17).

Aqueous nutrient release studies are essential for understanding the behaviour of fertilisers upon exposure to water, simulating scenarios such as irrigation, rainfall, soil moisture fluctuations and hydroponic systems (18). Environmental factors including pH, temperature and salinity significantly influence release dynamics. Acidic conditions accelerate matrix hydrolysis, whereas alkaline environments reduce nutrient solubility (19). Elevated temperatures promote polymer swelling and diffusion, with a 24 % increase in nitrogen release observed from starch-based CRFs between 15°C and 35°C (20). Similarly, increased salinity (2–4 dS m^{-1}) suppresses water uptake and diffusion, resulting in a 25 % reduction in potassium release (21). Release kinetics are commonly analysed using models such as zero-order, first order, Higuchi and Korsmeyer–Peppas, which facilitate interpretation of the underlying mechanisms governing nutrient transport (22). Lignin-based CRFs frequently exhibit non-Fickian release behaviour, driven by both diffusion and matrix relaxation, supporting predictive modeling and the development of environmentally compatible fertiliser systems (23).

However, most existing lignin-based controlled-release fertilisers primarily focus on single-nutrient systems or rely on simple physical encapsulation and coating approaches, which often lack structural stability and environmental responsiveness. Additionally, limited studies have explored simultaneous nitrogen and potassium incorporation within a chemically cross-linked lignosulfonate matrix, particularly using low-cost, biodegradable crosslinkers such as citric acid. The present formulation distinguishes itself by integrating dual nutrient loading (N and K) within a covalently and ionically stabilised lignosulfonate network, enabling controlled diffusion through a structurally reinforced matrix. Furthermore, unlike conventional lignin-based CRFs, this system is specifically designed to exhibit environment-responsive release behaviour under pH, temperature and salinity conditions, coupled with a comprehensive evaluation linking release kinetics with biological performance through hydroponic bioassay. This integrative approach provides a more realistic assessment of fertiliser behaviour across simulated environmental scenarios.

Despite progress, current research lacks integrative assessments linking polymer structure, nutrient release dynamics and eco-safety under realistic aqueous conditions. It is hypothesised that the citric acid cross-linked lignosulfonate matrix can effectively regulate nitrogen and potassium release through controlled diffusion and matrix interactions, while maintaining environmental compatibility under varying aqueous conditions. To address these gaps, the present study aims to: (i) compare nitrogen and potassium release profiles of a citric acid cross-linked lignosulfonate nitrogen–potassium (NK) biopolymer fertiliser with urea and muriate of potash (MOP) under varied aqueous pH, temperature and salinity conditions, (ii) evaluate nutrient release kinetics using multiple mathematical models and (iii) assess environmental safety through a hydroponic phytotoxicity assay.

Materials and Methods

Materials

All chemicals used were of analytical grade and employed without further purification. Prilled urea (46 % N), potassium chloride (60 % K_2O) and calcium lignosulfonate (technical grade, ≥90 % purity), were procured from HiMedia Laboratories Pvt. Ltd., Mumbai, India. Citric acid monohydrate (≥99.5 %) and sodium chloride (AR grade) were obtained from Sisco Research Laboratories (SRL), India.

Synthesis of citric acid crosslinked lignosulfonate NK biopolymer fertiliser

The nitrogen-potassium (NK) biopolymer fertiliser was synthesised by adapting protocols from previous report (24). Calcium lignosulfonate was dissolved in distilled water at 50±2°C under continuous stirring. Urea and potassium chloride were added at predetermined ratios as nitrogen and potassium sources, respectively and allowed to dissolve completely. The formulation was designed to obtain a final nutrient composition of 20:20 (N: K_2O basis) and the quantities of urea and potassium chloride were calculated accordingly. Citric acid, serving as a biodegradable crosslinking agent, was added gradually at 10–15 % (w/w of lignosulfonate) and the pH was adjusted to 4.5–5.0 using dilute NaOH or HCl to facilitate esterification. The mixture was maintained at 65–75 °C for 3 hr to promote covalent and ionic crosslinking. The resulting viscous mass was cast into trays and cured at 110–120°C for 2–3 hr in a hot air oven to ensure matrix stabilisation and moisture removal. The dried product was ground, sieved into uniform granules and stored in airtight containers for subsequent analyses. The structural and physicochemical characterisation of the synthesised biopolymer matrix has been conducted in a complementary study focusing on material development; therefore, the present work emphasises functional evaluation through nutrient release kinetics and biological assessment.

Aqueous nutrient release studies

Nutrient release was studied under varying environmental conditions, including pH (4.0, 7.0 and 9.0), temperature (25 and 35°C) and water type (distilled vs. saline water at 2 dS m^{-1}). In each test, 0.5 g of the biopolymer fertiliser was enclosed in a dialysis membrane and immersed in 100 mL of the respective solution. Conventional fertilisers (urea and MOP) were tested in nutrient-equivalent amounts. Samples were incubated under static conditions and 10 mL aliquots were withdrawn at each sampling interval and immediately replaced with an equal volume of fresh solution to maintain constant volume and sink conditions throughout the experiment. Ammonium (NH_4^-) concentrations were quantified using the indophenol blue method (λ=640 nm) and potassium (K^+) levels were measured by flame photometry (Systronics 128), following the method of previous researchers (25). Cumulative release percentage (E, %) was calculated using equation (1):

$$E = \sum_{i=1}^{n=1} \left[\frac{C_i \cdot V_e}{C_i \cdot V_f} + \frac{C_n \cdot (V_n - (n-1) . Ve)}{C_i \cdot V_f} \right] \text{———— (1)}$$

Where C_i and C_n are the nutrient concentrations (mg mL^{-1}) at the *i*th and *n*th sampling intervals, respectively; V_e is the volume of each aliquot (mL); V_0 is the initial volume of the release medium (mL); and V_f is the total nutrient content (mg). No direct swelling or degradation measurements were performed; mechanism interpretation was based on kinetic modelling.

Kinetic modelling

To elucidate the nutrient release mechanisms from the biopolymer fertiliser and conventional sources, cumulative nitrogen and potassium release data were fitted to 4 models: zero-order, first-order, Higuchi and Korsmeyer–Peppas. In the zero-order model, the release rate is constant and independent of nutrient concentration (26), described by the equation (2).

$$R_T = R_0 - C_0.T \qquad \text{———} (2)$$

where R_T is the cumulative amount of nutrient released at time t and C_0 is the zero-order release constant ($mghr^{-1}$). The first-order model assumes the release rate is proportional to the remaining nutrient concentration in the matrix (27) and is expressed as equation (3).

$$R_T = R_0 . e^{-C_1.T} \qquad \text{———} (3)$$

where C_1 is the first-order release constant (hr^{-1}).

The Higuchi model, suitable for diffusion-controlled release from planar matrices (28), is given by equation (4).

$$R_T = C_H . T^{1/2} \qquad \text{———} (4)$$

where C_H represents the Higuchi dissolution constant ($mgh^{-1/2}$).

The Korsmeyer–Peppas model, a semi-empirical approach widely used in polymeric systems (22), describes release as equation (5).

$$\frac{R_T}{R_0} = C_k . T^n \qquad \text{———} (5)$$

where R_T/R_0 is the fractional release at time t, C_k is the kinetic constant and n is the release exponent. The value of n is used to characterise the release behaviour, where $n \leq 0.45$ indicates Fickian diffusion, $0.45<n<0.89$ indicates anomalous transport, $n = 0.89$ corresponds to case II transport and $n>0.89$ represents super case II transport. Goodness of fit was evaluated based on the value of coefficient of determination (R^2).

Environmental safety evaluation

A hydroponic bioassay was conducted using *Zea mays* L. to assess environmental safety. Seeds were germinated in Petri dishes irrigated with aqueous leachates derived from three treatments: control (distilled water), conventional fertiliser and the biopolymer fertiliser. After 7 days, germination percentage, root and shoot lengths, seedling vigour index (SVI), relative root elongation and chlorophyll index were measured. Data were expressed as mean ± standard error (n = 5) and subjected to one-way ANOVA to test for treatment effects, followed by Tukey's HSD post-hoc test to separate means at $p<0.05$. This assay served as an ecologically relevant indicator of the fertiliser's phytotoxicity and compatibility with early plant development.

Results and Discussion

Nutrient release behaviour under aqueous conditions

Effect of pH

The nutrient release from the biopolymer fertiliser was strongly influenced by the pH of the medium (Fig. 1). At pH 7.0, nitrogen and potassium exhibited a gradual release profile, with N release reached 38.12 % and K release 34.76 % on day 10, followed by a steady increase to a maximum cumulative release of 93.73 % (N) and 83.42 % (K) by day 30. In contrast, under acidic conditions (pH 4.0), nitrogen and potassium release declined to 85.73 % and 77.30 %, respectively, whereas under alkaline conditions (pH 9.0), the release dropped further to 77.93 % (N) and 71.64 % (K). The reduced release under acidic conditions can be attributed to proton-induced contraction of the lignosulfonate matrix and reduced swelling, whereas in alkaline pH, the limited solubility of lignosulfonate and enhanced ionic crosslinking with citric acid hindered nutrient diffusion, despite possible hydrolysis of ester linkages (29). This behaviour suggests that pH not only affects matrix swelling but also alters the ionisation state of functional groups (–COOH and $–SO_3H$), thereby influencing electrostatic interactions within the polymer network and regulating nutrient mobility.

In contrast, conventional fertilisers (urea and MOP) exhibited a burst release pattern, with over 90 % nutrient solubilisation within 24 hr and nearly complete release (>99 %) by day 3 across all pH levels, reflecting their high solubility and lack of release control. The pronounced difference between the biopolymer and conventional fertilisers highlight the role of matrix-mediated diffusion resistance, where nutrient release is governed by both physicochemical interactions and structural constraints rather than simple dissolution. These observations align with previous reports, where pH variations modulated matrix swelling and degradation in biopolymer-based controlled-release fertilisers (30–32).

Effect of temperature

Temperature exerted a marked influence on the nutrient release behaviour (Fig. 2). At 35°C, the biopolymer fertiliser showed enhanced cumulative nitrogen and potassium release of 97.32 % and 89.12 % by day 30, respectively, compared to 89.60 % N and 79.32 % K at 25°C. This increase is attributed to enhanced polymer hydration, increased chain mobility and accelerated hydrolysis of ester bonds at elevated temperatures (33, 34). These effects facilitate matrix loosening and faster diffusion of nutrients, particularly during the initial phase. These results are consistent with the findings of previous researchers, who reported a 24 % increase in nitrogen release from starch-based controlled release fertiliser (CRF) between 15°C and 35°C (35) and support the findings of another study, highlighting temperature sensitivity of cross-linked polymer networks (36). Additionally, elevated temperature may accelerate the hydrolysis of ester linkages formed during citric acid crosslinking, contributing to partial matrix relaxation and increased permeability. In contrast to the biopolymer, conventional fertilisers showed over 96 % of nitrogen (urea) and 98 % of potassium (MOP) were released within 24 hr at 25°C, with only marginal acceleration at 35°C, reaffirming their immediate solubility without controlled-release mechanisms (37). This minimal temperature sensitivity further confirms that nutrient release from conventional fertilisers is dissolution-driven, whereas the biopolymer system exhibits diffusion-controlled and temperature-responsive behaviour.

Effect of water type

The type of water significantly impacted release behaviour (Fig. 3). In distilled water, the biopolymer exhibited a gradual and sustained release, reaching 91.46 % nitrogen and 81.23 % potassium by day 30. Release during the first 7 days was notably progressive, with nitrogen increasing from 9 % at 1 hr to 75 % by day 7, while in saline water (2 dS m^{-1} NaCl), the release decreased to 76.31 % N and 72.53 % K,

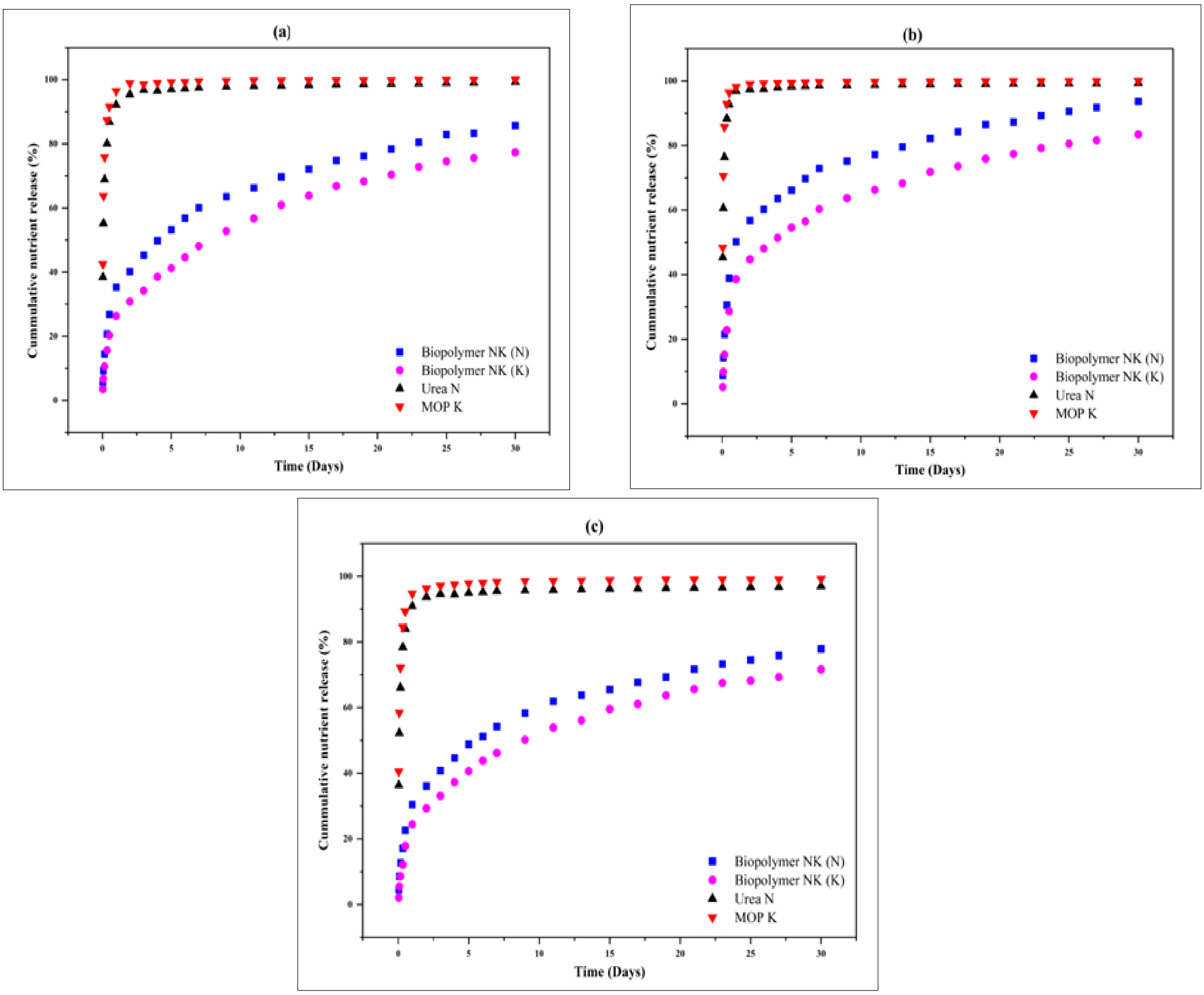

Fig. 1. Cumulative nutrient release (%) of nitrogen and potassium from the citric acid cross-linked lignosulfonate NK biopolymer fertiliser compared to urea and MOP under aqueous conditions at varied pH levels: (a) pH 4; (b) pH 7; (c) pH 9.

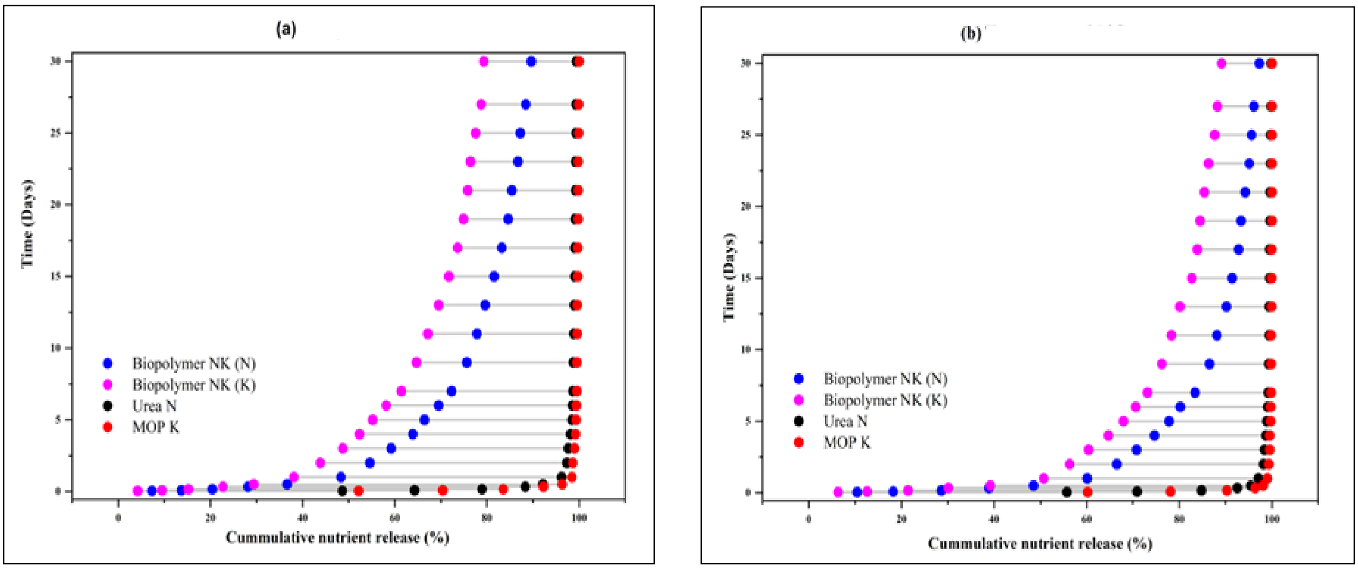

Fig. 2. Cumulative nutrient release (%) of nitrogen and potassium from the citric acid–crosslinked lignosulfonate NK biopolymer fertiliser compared with urea and MOP under aqueous conditions at two different temperatures: (a) 25 °C; (b) 35 °C.

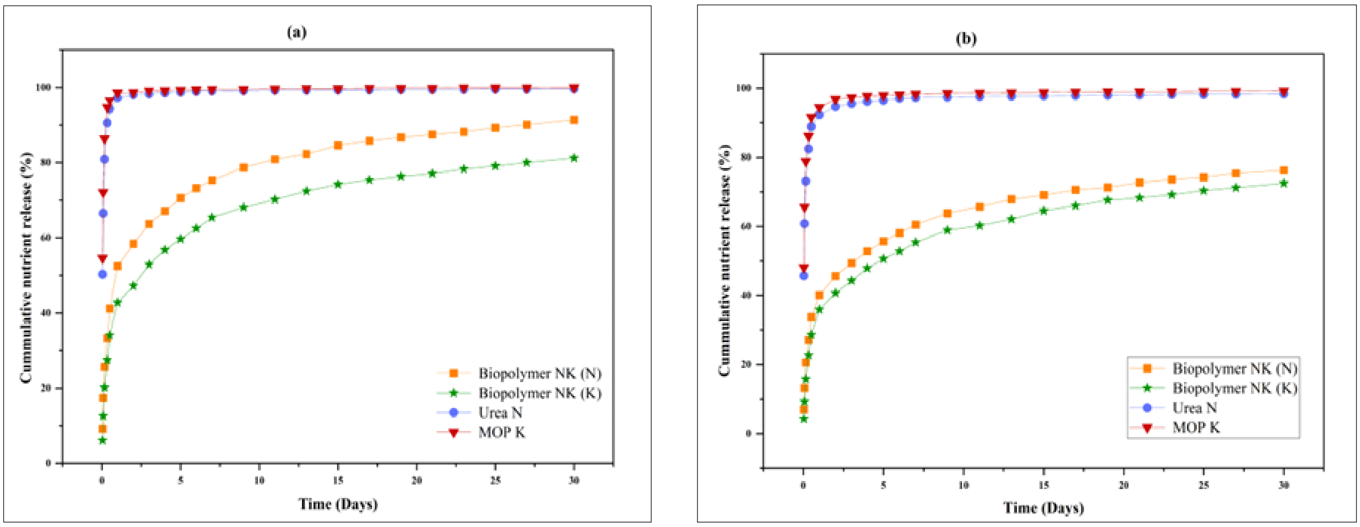

Fig. 3. Cumulative nutrient release (%) of nitrogen and potassium from the citric acid–crosslinked lignosulfonate NK biopolymer fertiliser compared to urea and MOP in: (a) distilled water; (b) saline water at $2\ dS\ m^{-1}$.

indicating a reduction of 12.46 % and 14.29 %, respectively. Salinity likely imposes osmotic resistance to water influx and reduces matrix swelling. Moreover, ionic interactions between Na^+ and sulfonic/carboxylic groups in lignosulfonate could further restrict polymer relaxation and nutrient migration (38, 39). The presence of competing ions in saline conditions may also reduce the effective diffusion gradient, thereby slowing nutrient release from the polymer matrix. These findings are consistent with earlier reports, that observed a 25 % reduction in K^+ release from bio-based fertilisers under saline conditions (40). This behaviour demonstrates the environmental responsiveness of the biopolymer system, which adapts its release characteristics based on external ionic conditions, unlike conventional fertilisers. In contrast, conventional fertilisers showed minimal differences between distilled and saline conditions, again reflecting their lack of environmental responsiveness.

Kinetic modelling of nutrient release

Kinetic modelling was performed to elucidate the nutrient release mechanisms of biopolymer NK fertiliser, urea and MOP under standard conditions (pH 7.0, 25°C, distilled water) (Fig. 4). Among the 4 models evaluated (Table 1), the Korsmeyer-Peppas model best described the release behaviour of the biopolymer, with high R^2 values for both nitrogen (0.91) and potassium (0.91). The corresponding release exponent (n) values were 0.79 and 0.72, respectively, indicating anomalous (non-Fickian) transport, where both diffusion and matrix relaxation contribute to nutrient release (27). This interpretation is further supported by the observed sensitivity of nutrient release to environmental factors such as pH, temperature and salinity, which influence both diffusion pathways and matrix dynamics. This behaviour is typical for hydrophilic polymeric matrices undergoing both swelling and erosion during release, as reported previously for lignin-based CRFs (8). In contrast, the conventional fertilisers exhibited best fit with the first-order model (R^2 >0.95), indicating a concentration-dependent, dissolution-controlled release, which aligns with the fast solubility of urea and MOP.

Environmental safety evaluation (Hydroponic bioassay)

The hydroponic assay revealed the environmental safety and bio efficacy of the synthesised biopolymer fertiliser (Fig. 5, Table 2). The germination rate in the biopolymer treatment was 96.2 ± 2.08 %, higher than both conventional (92.0±0.83 %) and control (88.6±1.64 %) treatments. The root length (13.1±0.26cm) and shoot length (11.3±0.34cm) in the biopolymer group surpassed the conventional group (9.2±0.34cm root, 8.8±0.42cm shoot) and control. Correspondingly, the seedling vigour index (SVI) was highest in the biopolymer group (1673.9), followed by conventional (1288.0) and control (1010.0). Notably, relative root growth (RRG) in the biopolymer treatment was 45.9 % higher than in the conventional treatment, while chlorophyll content (SPAD value) peaked at 39.8, indicating superior physiological performance.

These outcomes are attributed to the controlled nutrient release behaviour of the lignosulfonate matrix, which contains hydrophilic -COOH and $-SO_3H$ groups that limit solubility and enable gradual nutrient diffusion under aqueous conditions. This reduces osmotic shock and salt toxicity often seen with fast-releasing fertilisers like urea and MOP. The improved germination and seedling growth parameters can be directly linked to the sustained availability of nutrients, which ensures a balanced supply of nitrogen during early growth stages, thereby enhancing metabolic activity and chlorophyll

Table 1. Model-derived kinetic constants characterising nutrient release behaviour from biopolymer and conventional fertilisers

Models	Parameters	Biopolymer NK (N)	Biopolymer NK (K)	Urea N	MOP K
Zero-order	q0	453.03	372.07	880.81	-4.79
	k0	-20.54	-19.41	-5.84	904.84
	R^2	**0.63**	**0.67**	**0.18**	**0.14**
First-order	q0	387.94	309.87	863.65	891.29
	k1	-0.04	-0.04	-0.01	-0.005
	R^2	**0.414**	**0.41**	**0.91**	**0.90**
Higuchi	kH	132.52	123.99	43.15	35.63
	R^2	**0.84**	**0.87**	**0.34**	**0.29**
Korsmeyer-Peppas	k	0.44	0.39	0.86	0.88
	n	0.79	0.72	0.07	0.05
	R^2	**0.91**	**0.91**	**0.32**	**0.27**

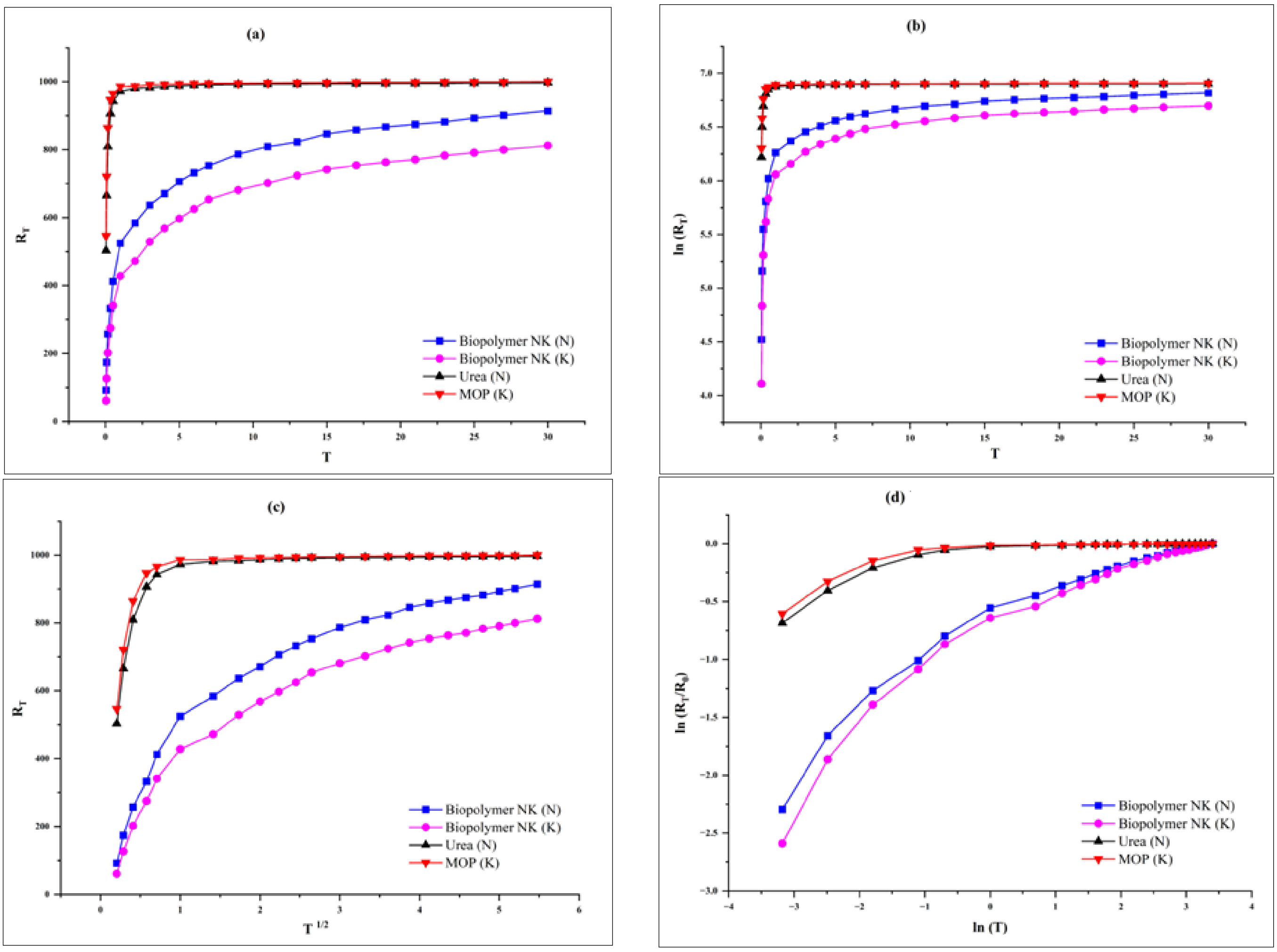

Fig. 4. Kinetic modelling of cumulative nitrogen and potassium release from the citric acid cross-linked lignosulfonate NK biopolymer fertiliser compared with urea and MOP using: (a) zero-order kinetics; (b) first-order kinetics; (c) Higuchi model; (d) Korsmeyer–Peppas model.

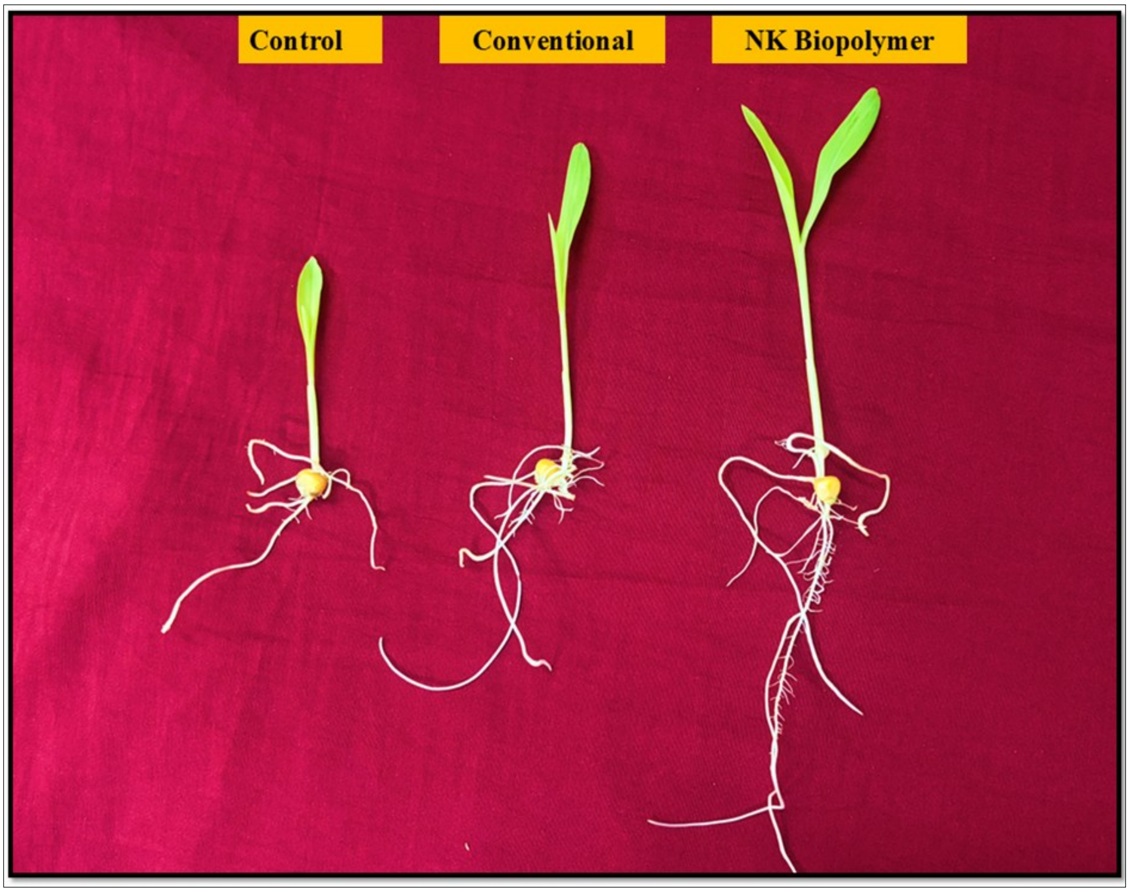

Fig. 5. Hydroponic bioassay evaluating the environmental safety and bio efficacy of NK biopolymer fertiliser using *Zea mays* seedlings.

Table 2. Hydroponic assay-based assessment of maize seedling response to fertilisers

Parameters	Control	Biopolymer	Conventional
Germination rate (%)	88.6 ± 1.64^{c}	96.2 ± 2.08^{a}	92 ± 0.8^{b}
Root length (cm)	8.3 ± 0.24^{c}	13.1 ± 0.26^{a}	9.2 ± 0.15^{b}
Shoot length (cm)	6.1 ± 0.09^{c}	11.3 ± 0.12^{a}	8.8 ± 0.15^{b}
Seedling vigour index (SVI)	1010 ± 8.01^{c}	1673 ± 11.2^{a}	1288 ± 13.4^{b}
% Relative root growth (%)	-	280 ± 8.9^{a}	90 ± 7.4^{b}
Chlorophyll index (SPAD)	30.6 ± 1.2^{b}	39.8 ± 0.9^{a}	32.4 ± 0.7^{ab}

Values are expressed as mean ± standard error (n = 5). Means followed by different letters within a row are significantly different at p<0.05 according to Tukey's HSD test.

synthesis. In contrast, rapid nutrient release from conventional fertilisers may create localised high salt concentrations, leading to osmotic stress and reduced root elongation, as reflected in the lower RRG and SVI values. Furthermore, lignosulfonate-based materials are reported to exhibit bioactive properties, including hormone-like (auxin-like) effects, which may contribute to enhanced root development and nutrient uptake efficiency. These results are consistent with earlier findings on lignin-based controlled-release systems, confirming the agronomic potential and environmental safety of such materials (41–44). However, as the present study is limited to aqueous and hydroponic systems, further validation under soil and field conditions is necessary to confirm their performance under practical agricultural environments.

Conclusion

The study demonstrates that the citric acid cross-linked lignosulfonate biopolymer enables controlled and sustained release of nitrogen and potassium, with release behaviour influenced by environmental conditions such as pH, temperature and salinity. Kinetic analysis indicates that nutrient release is governed by combined diffusion and matrix relaxation processes, resulting in improved nutrient delivery compared to conventional fertilisers. Hydroponic evaluation further indicates enhanced seedling growth and physiological performance under controlled conditions. Future studies should focus on soil incubation and field validation to confirm agronomic performance under practical agricultural conditions.

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Authors' contributions

MM carried out the experiments, analysed the data and wrote the draft manuscript. JD, SBK, RR, SA and BK conceived, designed and coordinated the experiments and revised the manuscript. All authors read and approved the final manuscript.

Compliance with ethical standards

Conflict of interest: Authors do not have any conflict of interests to declare.

Ethical issues: None

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the authors used ChatGPT to improve language and clarity. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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