



Design, Formulation, and Kinetic Evaluation of a Slow-Release NPK Fertilizer Derived from Engineered Biochar and Sewage Sludge

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ABSTRACT

Conventional mineral fertilizers exhibit poor nutrient use efficiency (<50% for N and P), driving environmental degradation through eutrophication and greenhouse gas emissions. This study reports the design, formulation, and mechanistic evaluation of a biochar-based slow-release fertilizer (BSRF) synthesized from ball-milled groundnut shell biochar and sewage sludge—two abundant waste streams. BSRF exhibited sustained release of N ($78 \pm 2.8\%$), P ($68 \pm 2.5\%$), and K ($72 \pm 2.6\%$) over 30 days, with time to 50% release (T_{50}) = 18.2 ± 1.2 to 18.9 ± 1.1 days) compared to 5.1–5.3 days for conventional NPK fertilizer ($p < 0.001$). Multi-model kinetic analysis (first-order, Higuchi, and Korsmeyer-Peppas) revealed Fickian diffusion-controlled release ($n = 0.40$ – 0.42 ; $R^2 > 0.98$), with first-order rate constants ($k_1 = 0.058$ – 0.068 day^{-1}) 3.5- to 4.2-fold lower than conventional fertilizer ($p < 0.01$). Agronomic evaluation demonstrated 19% higher maize shoot biomass (4.82 ± 0.31 vs. $4.05 \pm 0.28 \text{ g plant}^{-1}$; $p < 0.05$). Heavy metal concentrations (Pb: 38 ± 2 ; Cd: 0.9 ± 0.1 ; total Cr: $55 \pm 4 \text{ mg kg}^{-1}$) complied with EU Regulation 2019/1009 limits. This waste-derived composite demonstrates diffusion-controlled slow-release behavior, offering a scalable, low-cost alternative to polymer-coated formulations while advancing circular economy principles in sustainable agriculture.

INTRODUCTION

Global food production must increase by 70% by 2050, intensifying reliance on synthetic fertilizers. Annual global N, P, and K consumption exceeds 200 million tonnes and continues to rise. However, conventional fertilizer efficiency is low: 40–70% of N, 80–90% of P, and 50–70% of

K are lost through leaching, volatilization, and runoff. These losses cause approximately \$100 billion in annual economic losses and drive eutrophication, groundwater nitrate contamination, N_2O emissions, and soil acidification. The mismatch between rapid fertilizer dissolution and crop nutrient demand creates a “nutrient

gap,” necessitating multiple applications and increasing costs and environmental loading. Nitrogen is especially vulnerable through ammonia volatilization (10–40%), nitrate leaching (10–30%), and denitrification (10–20%), while phosphorus runoff triggers eutrophication and hypoxic zones. These issues have driven interest in slow-release (SRFs) and controlled-release fertilizers (CRFs). Commercial CRFs improve nutrient use efficiency by 20–50% but face major limitations. Most coatings are non-biodegradable, petroleum-based polymers that persist in soil and contribute to microplastic contamination (0.5–5.0 g kg⁻¹ after repeated applications). Production costs (\$800–\$1,200 per ton) also make them inaccessible to smallholder farmers, who produce 70% of global food. This creates a sustainability paradox in regions most vulnerable to food insecurity. There is therefore a need for biodegradable, low-cost nutrient carriers from renewable resources.

Biochar is a promising platform due to its high surface area (50–500 m² g⁻¹), porosity, oxygen-containing functional groups, and cation exchange capacity, enabling nutrient retention via complexation, ion exchange, electrostatic attraction, and physical entrapment. Biochar also offers carbon sequestration, improved soil properties, and reduced N₂O and CH₄ emissions. Groundnut shells are an abundant, underutilized feedstock in sub-Saharan Africa, with approximately 1.2 million tonnes generated annually; current disposal by burning or landfilling wastes a valuable resource. Pyrolysis valorizes these shells into biochar. However, pristine biochar has limited surface functionality and dispersibility. High-energy ball milling enhances surface area (>400 m² g⁻¹), functional group density, dispersibility, nutrient loading (30–50% higher), and release half-life (20–30% longer).

Sewage sludge, generated at over 120 million tonnes dry solids annually, is rich in N (3–6%), P (1–3%), K (0.5–1%), and micronutrients. Direct land application is limited by heavy metals, pathogens, and organic contaminants. Pyrolysis at ≥500°C reduces heavy metal bioavailability by 60–90%, eliminates pathogens and organic contaminants, and produces a nutrient-rich biochar suitable for soil application.

Integrating ball-milled biochar with sewage sludge offers a synergistic approach: valorizing agricultural and municipal wastes, enhancing nutrient use efficiency, reducing pollution, and providing affordable fertilization. Key knowledge gaps remain: the combination of ball-milled biochar and sewage sludge as a formulated NPK source is underexplored; multi-nutrient release kinetics are not comprehensively modelled; comparative evaluations with conventional fertilizers under standardized conditions are limited, especially for groundnut shells; and heavy metal safety assessments against international standards are often absent. No study has integrated ball-milled groundnut shell biochar with sewage sludge into a

formulated NPK fertilizer and systematically modelled its multi-nutrient release under standardized soil column conditions.

This study therefore aimed to: (i) develop a novel slow-release NPK fertilizer (BSRF) from ball-milled groundnut shell biochar and sewage sludge via scalable pelletization; (ii) characterize its physicochemical properties; (iii) evaluate 30-day N, P, and K release kinetics against conventional NPK using soil column leaching; (iv) elucidate release mechanisms via zero-order, first-order, Higuchi, and Korsmeyer-Peppas models; (v) assess maize shoot biomass as a proxy for agronomic performance; and (vi) quantify Pb, Cd, and total Cr against EU Regulation 2019/1009 limits. By transforming two waste streams into a value-added slow-release fertilizer, this work supports nutrient use efficiency, pollution reduction, smallholder affordability, circular economy principles, and SDGs 2, 6, 11, and 12.

MATERIALS AND METHODS

Materials

Analytical-grade chemicals (BDH, UK): KOH, NaOH, HCl, NaCl, H₃PO₄, urea, KCl, ammonium phosphate, and ammonium nitrate. Sewage sludge was collected from the wastewater treatment plant in Jos Metropolis, Nigeria; groundnut shells (*Arachis hypogaea* L.) were obtained from local farms. Deionized water was used throughout.

Equipment

Oven (105°C) for drying; muffle furnace (500°C, 2 h, N₂ flow 100 mL/min) for pyrolysis; planetary ball mill (10 mm zirconia balls, 20:1 ball-to-powder ratio, 300 rpm, 6 h with 10-min pauses every 30 min) for milling; pH meter (1:10 w/v suspension, 25 ± 2°C); FTIR spectrometer (4000–400 cm⁻¹, KBr pellet); XRD (Cu Kα, 10–80° 2θ); SEM (10–15 kV, gold-coated); soil incubation columns (30 cm × 5 cm diameter); and intelligent soil testing kit (STK-3000) for nutrient analysis.

Biochar Synthesis

Groundnut shells were washed, sun-dried (48 h), oven-dried (105°C, 24 h), crushed, ground, and sieved. Powder (~20 g) was pyrolyzed at 500°C for 2 h (ramp 10°C/min). Bulk biochar was KOH-activated (1:3 w/w biochar:KOH) at 500°C for 1 h, washed with 0.1 M HCl and deionized water to pH 7.0, dried, and ball-milled to nanobiochar under the conditions above.

Sewage Sludge Processing

Fresh sludge was settled for 3 days, supernatant decanted, air-dried (7 days), oven-dried (105°C, 24 h), ground, and sieved (100-mesh). It was sterilized at 120°C for 30 min, ball-milled under the same conditions as biochar, and stored in a desiccator.

Surface Area Estimation and pH Measurement

Surface areas of non-activated biochar, activated biochar, sewage sludge, starch, and BSRF were estimated by Sear's method: 0.5 g sample dispersed in 50 mL distilled water with 10 g NaCl and 10 mL 0.1 M HCl, stirred 5 min, then back-titrated with 0.1 M NaOH to pH 9.0. Surface area was calculated from titration data according to Sear's method. pH was measured in 1:10 w/v water suspension at $25 \pm 2^\circ\text{C}$. Equation 1:

$$S\left(\frac{\text{m}^2}{\text{g}}\right) = 32v - 0.89 \quad (1)$$

where v is the volume of NaOH (mL) required to reach pH 9.0, and 32 and 0.86 are empirical constants. Measurements were performed in triplicate. Fertilizer pH was measured in a 1:10 (w/v) water suspension after 30 min stirring at $25 \pm 2^\circ\text{C}$ using a calibrated pH meter.

Formulation of NPK Biochar-Based Slow-Release Fertilizer (BSRF)

BSRF was formulated to a nominal 15:15:15 N: P_2O_5 : K_2O ratio (w/w/w). Activated nanobiochar and milled sewage sludge were combined at 1:2 (w/w). Urea, ammonium nitrate, potassium sulfate, and 85% orthophosphoric acid were dry-mixed with the biochar-sludge matrix in stoichiometric proportions. Sweet potato starch (5–10% w/w) was added as a biodegradable binder. The mixture was heated (70 – 80°C) for starch gelatinization, oven-dried (50°C , 12 h), granulated, and sieved (1–2 mm). Granules were pressed into cylindrical pellets (hydraulic press, 5 tons, 30 s), cured (50°C , 24 h), and stored airtight as BSRF. Actual N, P, and K concentrations were determined colorimetrically in triplicate using the STK-3000 kit. Nutrient loading efficiency (LE, %) was calculated using Equation 2:

$$LE (\%) = \frac{C_{\text{actual}} \times M_{\text{final}}}{C_{\text{theoretical}} \times M_{\text{input}}} \times 100 \quad (2)$$

where C_{actual} is the measured nutrient concentration in BSRF (% w/w), M_{final} is the mass of final BSRF product (g), $C_{\text{theoretical}}$ is the calculated nutrient concentration based on stoichiometric input (% w/w), and M_{input} is the total mass of nutrient-bearing inputs (g).

Structural and Morphological Characterization

FTIR spectra were recorded (Shimadzu FTIR-8400S, 4000 – 400 cm^{-1} , KBr pellet, 4 cm^{-1} resolution, 32 scans). XRD patterns were obtained (Bruker D8 Advance, Cu K α , $\lambda = 0.15406 \text{ nm}$, 40 kV, 40 mA, 2θ 10 – 80° , step 0.02°). Morphology was examined by SEM (Phenom ProX, 10 – 15 kV , gold-coated).

Soil Incubation and Nutrient Release Dynamics

Soil columns ($30 \text{ cm} \times 5 \text{ cm}$ i.d.) were constructed from transparent polypropylene cylinders. BSRF and conventional fertilizer (CF) were applied at equivalent NPK rates. Incubation was at $25 \pm 2^\circ\text{C}$. Leachates were collected on days 1, 3, 7, 14, 21, and 28, and analyzed for

N, P, and K using the STK-3000 kit. Cumulative release was calculated as the percentage of total applied nutrients recovered.

Kinetic Analysis

Nutrient release kinetics were modelled using four equations:

$$\text{Zero-order model: } Q_t = Q_0 + k_0 t \quad (3)$$

$$\text{First-order model: } \ln(Q_\infty - Q_t) = \ln Q_\infty - k_1 t \quad (4)$$

$$\text{Higuchi model: } Q_t = k_H \sqrt{t} \quad (5)$$

$$\text{Korsmeyer-Peppas-Model: } Q_t/Q_\infty = K_p t^n \quad (6)$$

where Q_t is the cumulative release at time t , Q_0 is the initial release, Q_∞ is the total release at infinite time, k_0 is the zero-order rate constant, k_1 is the first-order rate constant, k_H is the Higuchi rate constant, K_p is the Korsmeyer-Peppas constant, and n is the release exponent. For cylindrical geometries, $n < 0.45$ indicates Fickian diffusion; $n = 0.45$ indicates Case II transport; and $0.45 < n < 1.0$ indicates anomalous transport. Only initial-release data ($\leq 60\%$ cumulative release) were used to calculate n -value. T_{50} values were calculated by interpolation from first-order model fits.

Agronomic Performance Evaluation

Maize (*Zea mays* L) was grown in soil columns ($30 \text{ cm} \times 5 \text{ cm}$) packed with air-dried, sieved agricultural soil (bulk density $\sim 1.2 \text{ g cm}^{-3}$). Initial soil properties: pH 6.2, total N 0.08% , available P 12 mg kg^{-1} (Bray-1), exchangeable K 85 mg kg^{-1} . Seeds were surface-sterilized ($1\% \text{ NaOCl}$, 5 min), sown at 2–3 cm depth, and thinned to one plant per column. Treatments ($n=3$): (i) unfertilized control, (ii) CF at 120 kg N ha^{-1} , $60 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$, and $60 \text{ kg K}_2\text{O ha}^{-1}$, and (iii) BSRF at equivalent NPK rates. Fertilizers were applied after germination and shoot biomass was measured after 3 weeks.

Heavy Metal Analysis

Sewage sludge and BSRF were digested in aqua regia (3:1 HCl: HNO_3) at 120°C for 2 h and analyzed by atomic absorption spectroscopy (AAS, PerkinElmer Optima 8000) for Pb, Cd, and total Cr. Detection limits: Pb 0.05 mg/kg , Cd 0.01 mg/kg , total Cr 0.10 mg/kg . Results were compared to EU Fertilizer Regulation 2019/1009 limits (Pb: 120, Cd: 1.5, total Cr: 200 mg kg^{-1}).

Statistical Analysis

All experiments were conducted in triplicate ($n = 3$), with results expressed as mean $\pm$ SD. BSRF and conventional fertilizer (CF) groups were compared using two-tailed Student's t -tests, assuming unequal variances (Welch's t -test) where appropriate. For multiple comparisons across time points and nutrients, Bonferroni correction controlled the family-wise error rate, with significance set at $p < 0.05$. Normality was assessed by Shapiro–Wilk test ($p > 0.05$ for all datasets), and variance homogeneity was verified by

Levene's test. Analyses were performed using GraphPad Prism version 9.5.0 (GraphPad Software, San Diego, CA, USA) and R version 4.2.1 (R Core Team, 2022).

RESULTS AND DISCUSSION

Physicochemical Characterization

pH Measurement

BSRF exhibited a pH of 7.2 ± 0.2 (1:10 w/v water suspension, 25 ± 2 °C, 30 min stirring). Corresponding pH values of precursor materials were: non-activated biochar 8.1 ± 0.3 , activated biochar 6.8 ± 0.2 , sewage sludge 6.5 ± 0.3 , and conventional fertilizer 5.8 ± 0.2 .

Structural and Morphological Characterization (FTIR, XRD, SEM)

FTIR, XRD and SEM results for BSRF and its precursors are presented in Figure 1. FTIR spectra (Figure 1a) displayed characteristic bands at ~ 3350 cm^{-1} (O–H, N–H), ~ 2900 cm^{-1} (aliphatic C–H), ~ 1630 cm^{-1} (C=O / aromatic C=C), ~ 1425 cm^{-1} (NO_3^-) and $1100\text{--}800$ cm^{-1} (P–O). XRD patterns (Figure 1b) showed a broad amorphous carbon halo ($2\theta = 20\text{--}30^\circ$) together with crystalline reflections attributable to struvite (15.8° , 20.9° , 30.1°), KCl (28.3° , 40.5° , 50.2°) and $(\text{NH}_4)_3\text{PO}_4$ (16.2° , 21.5° , 32.8°). SEM images revealed irregular, angular nanobiochar particles (Figure 1c–e), a flaky layered morphology for sewage sludge (Figure 1f–g), and a porous, heterogeneous composite structure for BSRF (Figure 1h–i) containing interconnected pore networks and interstitial spaces.

Nutrient Release Kinetics

Comparative Release Profiles

Cumulative N, P and K release over 30 days is summarised in Figure 2 and Table 1. BSRF released 78 ± 2.8 % N, 68 ± 2.5 % P and 72 ± 2.6 % K by day 30, with no pronounced initial burst (day-7 release: 28 ± 2.1 % N, 22 ± 1.8 % P, 25 ± 1.9 % K). Conventional fertilizer released substantially higher fractions by day 7 (65 ± 3.2 % N, 58 ± 2.9 % P, 62 ± 2.7 % K) and reached 92 ± 1.5 % N, 88 ± 1.8 % P and 90 ± 1.6 % K by day 30. Differences between the two fertilizers were statistically significant at both day 7 and day 30 for all three nutrients ($p < 0.001$). Time to 50 % release (T_{50}) derived from first-order fits was 18.2–18.9 days for BSRF versus 5.1–5.3 days for conventional fertilizer ($p < 0.001$).

Kinetic Modelling of Nutrient Release

Korsmeyer–Peppas Model

Korsmeyer–Peppas parameters are listed in Table 2. BSRF yielded release exponents $n = 0.40\text{--}0.42$ ($R^2 = 0.989\text{--}0.992$) and rate constants $K_p = 0.108\text{--}0.124$. Conventional fertilizer gave lower n values ($0.26\text{--}0.28$; $R^2 = 0.948\text{--}0.952$) and higher K_p values ($0.324\text{--}0.356$; $p < 0.01$).

First-Order Kinetic Analysis

First-order parameters appear in Table 3. BSRF rate constants were $k_1 = 0.058\text{--}0.068$ day^{-1} ($R^2 = 0.989\text{--}0.994$) with equilibrium capacities $Q_\infty = 71.5\text{--}81.2$ %. Conventional fertilizer exhibited 3.5- to 4.2-fold higher k_1 values ($0.212\text{--}0.245$ day^{-1} ; $p < 0.01$) and higher Q_∞ ($91.2\text{--}95.6$ %).

Higuchi Square-Root Model

Higuchi parameters are given in Table 4. BSRF produced excellent linear fits ($R^2 > 0.98$) with rate constants $k_H = 12.5\text{--}14.2$ % $\cdot\text{day}^{-1/2}$. Conventional fertilizer showed higher k_H values ($22.1\text{--}24.6$ % $\cdot\text{day}^{-1/2}$) and slightly lower coefficients of determination.

Zero-Order Kinetic Analysis

Zero-order parameters are summarised in Table 5. BSRF gave moderate-to-good fits ($R^2 = 0.9285\text{--}0.9874$) with $k_0 = 2.12\text{--}2.45$ % $\cdot\text{day}^{-1}$. Conventional fertilizer produced poorer fits ($R^2 = 0.8373\text{--}0.9041$) and higher k_0 values ($3.45\text{--}3.82$ % $\cdot\text{day}^{-1}$).

Agronomic Performance

After four weeks of growth, maize shoot biomass was 4.82 ± 0.31 g plant^{-1} under BSRF versus 4.05 ± 0.28 g plant^{-1} under conventional fertilizer (19 % higher; $p < 0.05$) and higher still relative to the unfertilized control (Figure 6). BSRF-treated plants also displayed darker green foliage.

Heavy Metal Analysis

Heavy-metal concentrations in sewage sludge and the finished BSRF are reported in Table 6. Relative to the sludge feedstock, BSRF contained lower levels of Pb (38 ± 2 vs 42 ± 3 mg kg^{-1}), Cd (0.9 ± 0.1 vs 1.2 ± 0.1 mg kg^{-1}) and total Cr (55 ± 4 vs 68 ± 5 mg kg^{-1}). All three metals remained well below the EU Regulation 2019/1009 limits for organic fertilizers (Pb 120, Cd 1.5, total Cr 200 mg kg^{-1}). Cr(VI) was not determined.

Discussion

pH and Structural Properties

The near-neutral pH of BSRF (7.2 ± 0.2) lies within the optimal range for most agricultural soils ($6.0\text{--}7.5$) and therefore minimises the risk of soil acidification or alkalinisation that can accompany repeated fertilizer applications (Zhang et al., 2005).

FTIR spectra confirmed successful incorporation of N-, P- and K-bearing species into the biochar–sludge matrix, as evidenced by the diagnostic NO_3^- and P–O bands together with the residual O–H/N–H and aromatic C=C features of the carbon skeleton (Zhang et al., 2015; Muhmood et al., 2019; Das & Ghosh, 2023). XRD further established the coexistence of an amorphous carbon phase with crystalline struvite, KCl and ammonium phosphate, indicating that a fraction of the nutrients is present as

discrete mineral phases that can dissolve more slowly than free soluble salts. SEM images of the finished pellets revealed a porous, heterogeneous microstructure containing interconnected pores and interstitial voids.

These physical features provide extended diffusion pathways and physical entrapment sites that are consistent with the retarded release kinetics observed experimentally (Das & Ghosh, 2023).

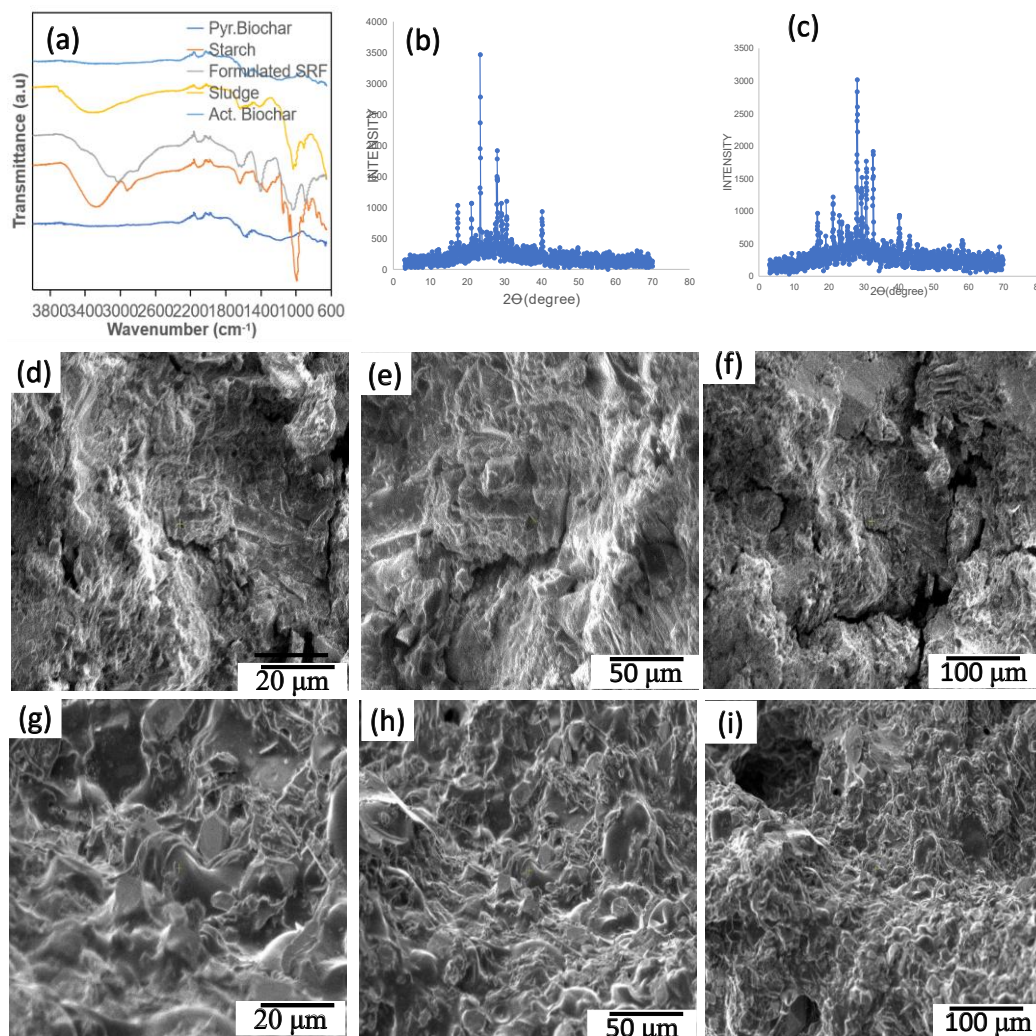


Figure 1: Structural and morphological characterization of precursor materials and BSRF composite. (a) FTIR spectra showing functional groups (O–H, N–H, C=O, NO₃⁻, P–O); (b) XRD patterns showing amorphous carbon and crystalline phases (struvite, KCl, (NH₄)₃PO₄); (c–e) SEM of nanobiochar (irregular, angular particles); (f–g) SEM of sewage sludge (flaky, layered morphology); (h–i) SEM of BSRF composite (porous, heterogeneous structure with embedded nanoparticles). Scale bars: 20–100 μm

Nutrient Release Behavior and Mechanisms

The release profiles of BSRF and conventional fertilizer were markedly different. Conventional fertilizer displayed the classic biphasic “burst-then-plateau” pattern expected of highly soluble NPK salts, with more than half of each nutrient liberated within the first week. In contrast, BSRF released nutrients gradually and continuously,

reaching only 22–28 % of total content by day 7 and 68–78 % by day 30. Consequently, T₅₀ values for BSRF were three- to four-fold longer than those of the conventional product. The absence of an initial burst and the sustained subsequent release are characteristic of a diffusion- or matrix-barrier-controlled system rather than simple dissolution.

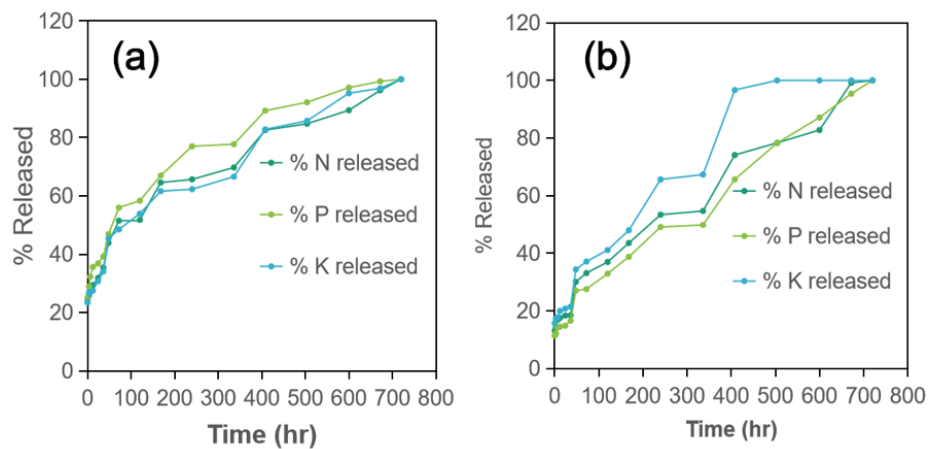


Figure 2: Cumulative nutrient release profiles over 30 days. (a) BSRF; (b) conventional fertilizer

Table 1: Comparative Summary of Cumulative Nutrient Release (%) (Mean $\pm$ SD, n=3)

Parameter	BSRF	Conventional Fertilizer
Nitrogen		
Day 7 release (%)	28 $\pm$ 2.1	65 $\pm$ 3.2***
Day 30 release (%)	78 $\pm$ 2.8	92 $\pm$ 1.5***
T ₅₀ (days)	18.2 $\pm$ 1.2	5.1 $\pm$ 0.6***
Phosphorus		
Day 7 release (%)	22 $\pm$ 1.8	58 $\pm$ 2.9***
Day 30 release (%)	68 $\pm$ 2.5	88 $\pm$ 1.8***
T ₅₀ (days)	18.2 $\pm$ 1.2	5.1 $\pm$ 0.6***
Potassium		
Day 7 release (%)	25 $\pm$ 1.9	62 $\pm$ 2.7***
Day 30 release (%)	72 $\pm$ 2.6	90 $\pm$ 1.6***
T ₅₀ (days)	18.9 $\pm$ 1.1	5.3 $\pm$ 0.4***

*Release percentages represent fraction of measured total nutrient content. T₅₀ values calculated by interpolation from first-order model fits. *** $p < 0.001$ vs. BSRF (two-tailed Student's *t*-test with Bonferroni correction).

Korsmeyer–Peppas Model Analysis

The Korsmeyer–Peppas release exponents obtained for BSRF ($n = 0.40\text{--}0.42$) fall below the threshold of 0.45 that marks Fickian diffusion for cylindrical geometries. This indicates that nutrient transport is governed primarily by diffusion through the porous biochar–sludge matrix. The corresponding K_p values were approximately three-fold lower than those of conventional fertilizer, confirming the retarding effect of the composite matrix. Phosphorus

exhibited the lowest K_p , consistent with additional retention via ligand exchange with surface functional groups on the biochar. The n values observed here are comparable to those reported for other rigid, non-swelling controlled-release matrices (Wu & Liu, 2008; Irfan et al., 2018) and are distinctly lower than the anomalous-transport exponents typical of swelling hydrogel coatings (Jamnongkan & Kaewpirom, 2010; Sempeho et al., 2014).

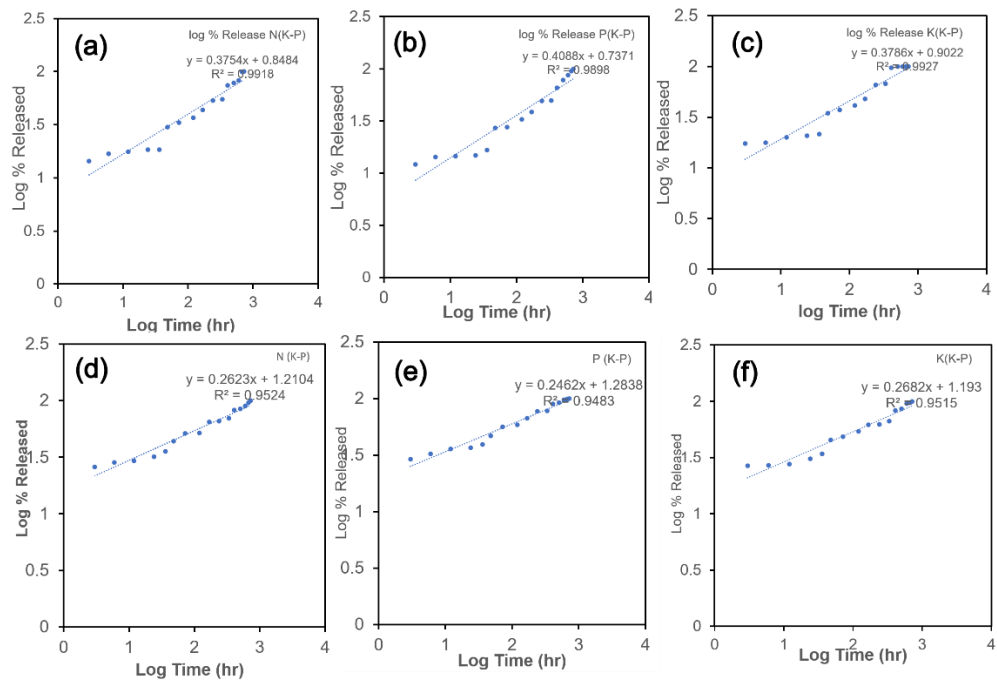


Figure 3: Korsmeyer-Peppas model fitting. (a–c) BSRF; (d–f) conventional fertilizer

Table 2: Korsmeyer-Peppas Model Parameters

Fertilizer Type	Nutrient	K _p	n	R ²	Release Mechanism	p-value (K _p)
BSRF	N	0.124	0.42	0.9918	Fickian diffusion	—
BSRF	P	0.108	0.40	0.9898	Fickian diffusion	—
BSRF	K	0.116	0.41	0.9927	Fickian diffusion	—
Conventional	N	0.356	0.28	0.9524	Fickian diffusion	<0.01
Conventional	P	0.324	0.26	0.9483	Fickian diffusion	<0.01
Conventional	K	0.338	0.27	0.9515	Fickian diffusion	<0.01

All $n < 0.45$ confirm Fickian diffusion for cylindrical pellet geometry. Conventional K_p significant ($p < 0.01$); BSRF p -values not reported as no significant difference was detected among replicates

First-Order Kinetic Analysis

First-order kinetics provided excellent descriptions of BSRF release ($R^2 \geq 0.989$). The rate constants (0.058–0.068 day⁻¹) were 3.5- to 4.2-fold lower than those of conventional fertilizer, and the asymptotic release capacities Q_∞ were also lower. These findings indicate

both slower desorption kinetics and stronger overall nutrient retention within the BSRF matrix, in agreement with earlier studies on biochar-based slow-release formulations (Yao et al., 2013; Lü et al., 2016; Ding et al., 2016; Agegnehu et al., 2017; Manikandan & Subramanian, 2021).

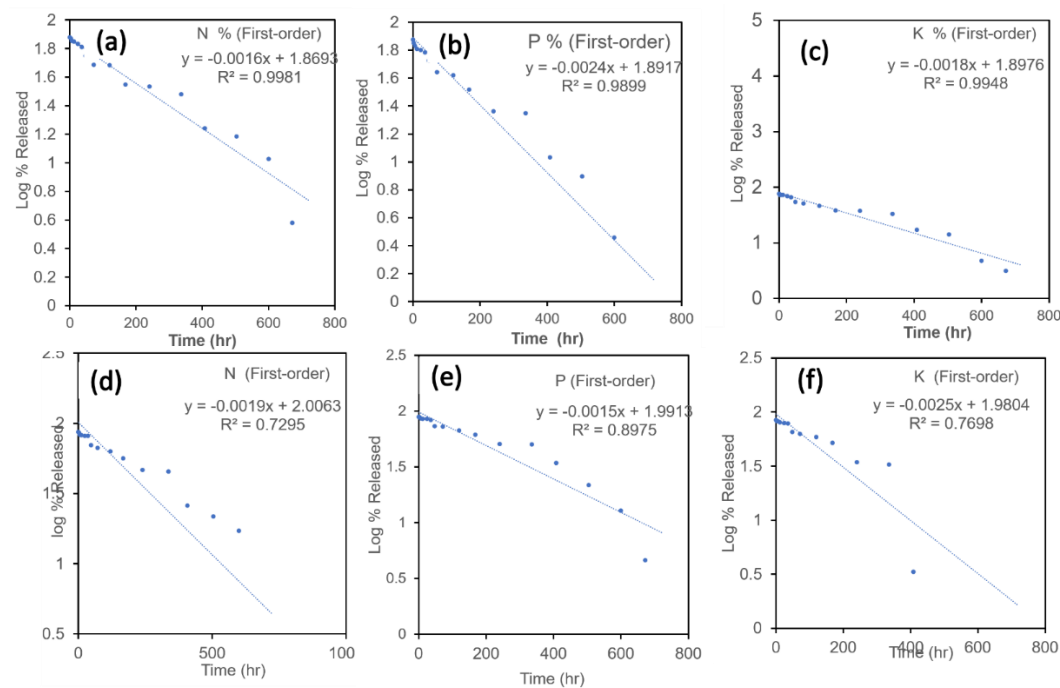


Figure 4: First-order kinetic model analysis. (a–c) BSRF; (d–f) conventional fertilizer

Table 3: First-Order Kinetic Model Parameters

Fertilizer Type	Nutrient	k_1 (day ⁻¹)	Q_∞ (%)	R^2	p-value (k_1)
BSRF	N	0.068	81.2	0.9981	—
BSRF	P	0.058	71.5	0.9899	—
BSRF	K	0.062	75.8	0.9948	—
Conventional	N	0.245	95.6	0.7295	<0.01
Conventional	P	0.212	91.2	0.8975	<0.01
Conventional	K	0.228	93.4	0.7698	<0.01

BSRF k_1 were significantly lower than conventional for all nutrients ($p < 0.01$)

Higuchi Square-Root Model Analysis

The Higuchi square-root model (Figure 5; Table 4) further supported diffusion-controlled release for BSRF, yielding excellent fits ($R^2 > 0.9997$ – 0.9975). Higuchi rate constants (k_H) for BSRF were 14.2 (N), 12.5 (P), and 13.4 %·day^{-1/2} (K). Conventional fertilizer showed poorer fits ($R^2 < 0.9907$ –

0.9975) and higher k_H values (22.1–24.6 %·day^{-1/2}), confirming its faster initial release. These results are consistent with prior reports (Siepmann & Peppas, 2011; Chen et al., 2018; Lateef et al., 2019; Liang et al., 2020; Sim et al., 2024).

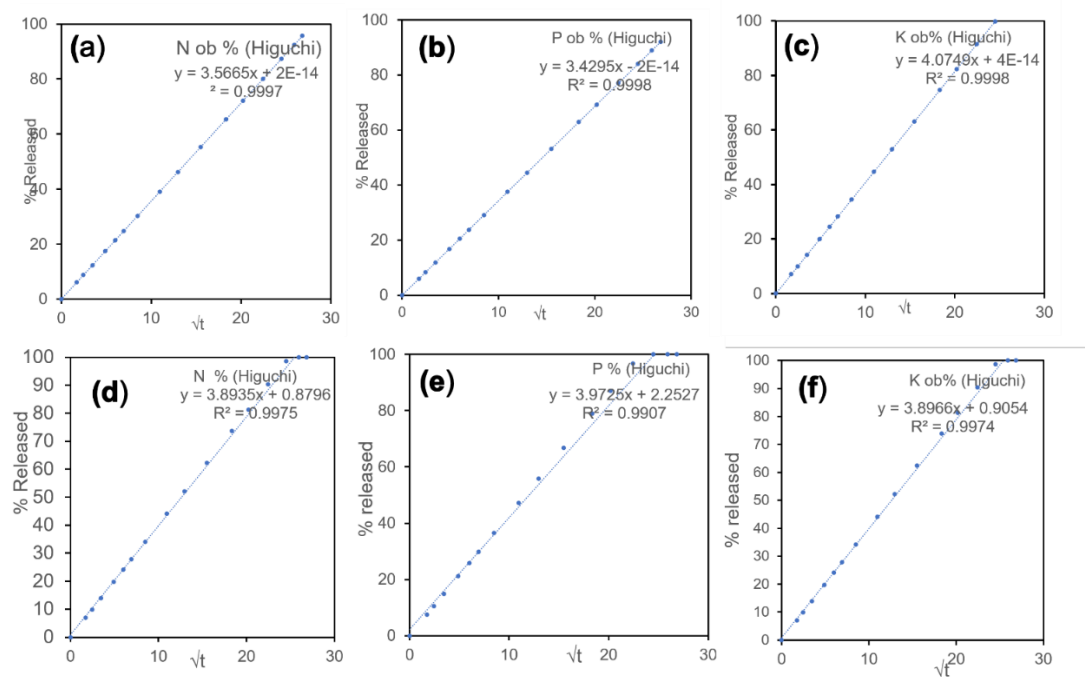


Figure 5: Higuchi square-root model analysis of nutrient release. (a–c) BSRF: nitrogen, phosphorus, and potassium; (d–f) conventional fertilizer: nitrogen, phosphorus, and potassium. Data points error represent mean $\pm$ SD (n = 3).

Table 4: Higuchi Square-Root Model Parameters

Fertilizer Type	Nutrient	k_H (%·day ⁻¹ /²)	R^2	p-value (k_H)
BSRF	N	14.2	0.9997	—
BSRF	P	12.5	0.9998	—
BSRF	K	13.4	0.9998	—
Conventional	N	24.6	0.9975	<0.01
Conventional	P	22.1	0.9907	<0.01
Conventional	K	23.2	0.9975	<0.01

BSRF k_H values were significantly lower than conventional for all nutrients ($p < 0.01$)

Zero-Order Kinetic Analysis

Zero-order kinetics provided only moderate fits for BSRF and poor fits for conventional fertilizer. The relatively low k_0 values of BSRF are consistent with gradual release, yet the non-significant p-values and incomplete model fit argue against pure surface-erosion control. Taken together with

the superior first-order, Higuchi and Korsmeyer–Peppas results, the data indicate that diffusion through a functionalized porous matrix is the dominant release mechanism, with possible secondary contributions from desorption or limited matrix erosion (Lü et al., 2016; Zhang et al., 2022).

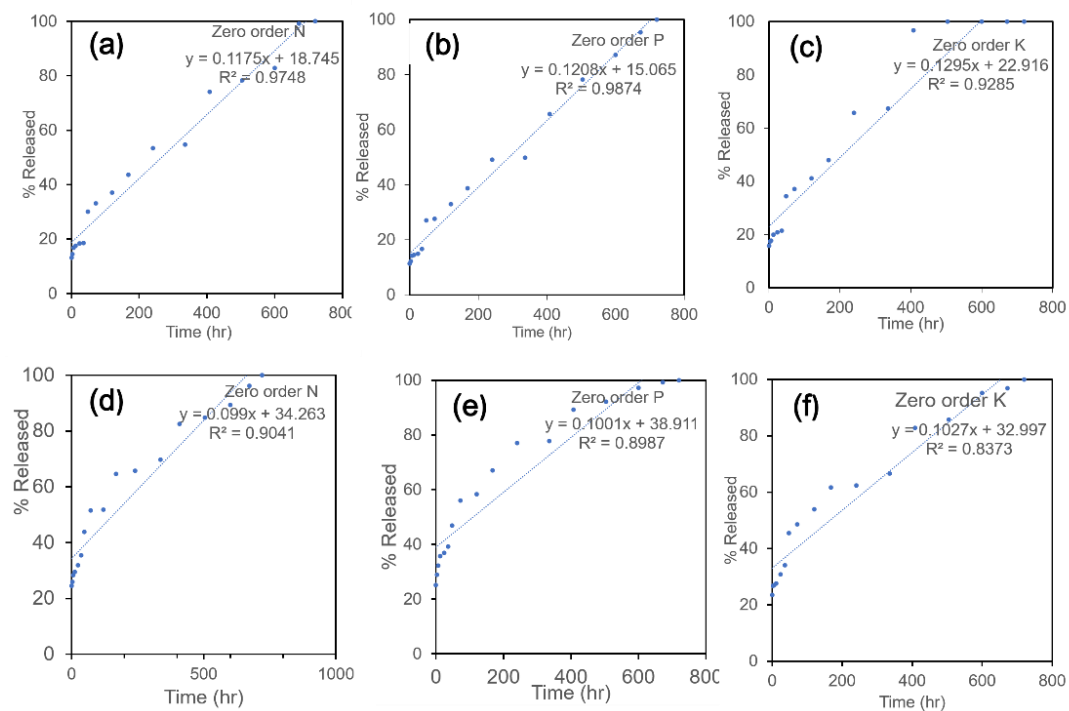


Figure 6: Zero-order kinetic model analysis for (a) nitrogen, (b) phosphorus, and (c) potassium. Moderate-to-good fits for BSRF ($R^2 = 0.9748\text{--}0.9285$) and poor fits for conventional fertilizer ($R^2 = 0.9041\text{--}0.8373$) rule out surface erosion as the dominant release mechanism. Data points represent mean $\pm$ SD ($n = 3$).

Table 5: Zero-Order Kinetic Model Parameters

Fertilizer Type	Nutrient	k_0 (%·day ⁻¹)	R^2	p-value (k_0)
BSRF	N	2.45	0.9748	—
BSRF	P	2.12	0.9874	—
BSRF	K	2.28	0.9285	—
Conventional	N	3.82	0.9041	<0.05
Conventional	P	3.45	0.8987	<0.05
Conventional	K	3.61	0.8373	<0.05

BSRF showed lower k_0 (2.45:2.12:2.28 %·day⁻¹) and higher R^2 (0.9285–0.9874) than the conventional fertilizer (3.82:3.45:3.61 %·day⁻¹; $R^2 = 0.8373\text{--}0.9041$). Nonsignificant p-values for BSRF k_0 , versus significant values ($p < 0.05$) for the conventional formulation, call the zero-order fit for BSRF into question. The moderate fits and statistical limitations suggest that zero-order kinetics are incomplete and that diffusion or other mechanisms contribute.

Agronomic Performance

The 19 % increase in maize shoot biomass and the darker green foliage observed under BSRF relative to conventional fertilizer are consistent with the prolonged nutrient-supply window ($T_{50} \approx 18\text{--}19$ days). By moderating the early burst and maintaining higher residual nutrient availability during the vegetative phase, the biochar–sludge composite appears to improve synchronisation between nutrient supply and crop demand. These agronomic benefits align with previous reports that slow-release fertilizers derived from organic wastes enhance early growth and nitrogen-use efficiency while reducing leaching losses (Agegnehu et al., 2017; Naz et al., 2021).

Heavy Metal Safety

All measured heavy-metal concentrations in the finished BSRF remained well below the EU Regulation 2019/1009 limits for organic fertilizers. Modest reductions relative to the raw sewage sludge are attributable to dilution by the biochar and nutrient additives. The values are comparable to, or lower than, those reported for other sludge- or biochar-based fertilizers (Wang et al., 2017; Zielińska & Oleszczuk, 2015) and satisfy European Biochar Certificate guidance (EBC, 2020). Because the EU regulation for organo-mineral fertilizers also requires Cr(VI) data, determination of hexavalent chromium is recommended in future work.

Table 6: Heavy Metal Concentrations in Sewage Sludge and BSRF (mg/kg dry weight)

Metal	Sewage Sludge	BSRF	EU Limit (2019/1009)
Pb	42 ± 3	38 ± 2	120
Cd	1.2 ± 0.1	0.9 ± 0.1	1.5
Total Cr	68 ± 5	55 ± 4	200

All values are within regulatory limits for organic fertilizers. Cr(VI) was not measured; recommended for future work

Mechanistic Role of Nanobiochar and Sewage Sludge in Controlled Release

Multi-model kinetic analysis converges on a coherent picture: BSRF release is governed by Fickian diffusion through a functionalized porous matrix. Nanobiochar contributes high surface area, oxygen-containing functional groups (carboxyl, hydroxyl, phenolic) that enable ion exchange, hydrogen bonding and electrostatic retention, and a network of pores that physically impedes nutrient diffusion. Sewage sludge supplies additional N, P and K while further increasing composite porosity and surface functionality. The net result is a biodegradable, waste-derived matrix that retards nutrient release without the need for synthetic polymer coatings.

Summary of Release Characteristics

Table 7 summarises the contrasting release behaviour of the two fertilizers. BSRF is characterised by Fickian diffusion control, first-order and Korsmeyer–Peppas kinetics as the best descriptors, sustained release over >30 days, high nutrient retention and consequently lower estimated leaching risk. Conventional fertilizer is dominated by rapid dissolution, a pronounced burst effect and a short effective feeding window (7–10 days). These differences underpin the agronomic and environmental advantages of the biochar–sludge composite.

Table 7: Summary of Release Mechanisms and Performance Indicators

Parameter	BSRF	Conventional
Primary release mechanism	Fickian diffusion	Rapid dissolution
Best-fit model(s)	First-order/ Korsmeyer–Peppas	First-order
Release rate classification	Slow and sustained	Fast with burst effect
Effective release duration	>30 days	7–10 days
Nutrient retention potential	High	Low
Estimated leaching risk	Reduced	High
Agronomic suitability	Extended feeding	Immediate but short-lived

Implications for Agricultural Sustainability

BSRF offers four practical sustainability benefits: (i) improved nutrient-use efficiency through sustained release matched to crop demand; (ii) reduced risk of nitrate leaching, ammonia volatilisation and phosphorus runoff; (iii) simultaneous valorisation of two abundant waste streams (groundnut shells and sewage sludge); and (iv) elimination of non-biodegradable polymer coatings that contribute to microplastic pollution. Because the formulation relies on locally available residues and simple pelletisation, production costs are expected to be substantially lower than those of commercial polymer-coated fertilizers, improving accessibility for smallholder farmers. Field-scale validation, long-term residual nutrient effects and full Cr(VI) speciation remain important next steps.

CONCLUSION

This study successfully designed and characterized a slow-release NPK fertilizer (BSRF) from ball-milled groundnut shell biochar and sewage sludge. BSRF demonstrated sustained nutrient release (N: 78%, P: 68%, K: 72% over 30 days) with T_{50} values 3–4 times longer than

conventional fertilizer ($p < 0.001$). Multi-model kinetic analysis confirmed Fickian diffusion-controlled release ($n = 0.40\text{--}0.42$; $R^2 > 0.98$), with first-order rate constants 3.5–4.2 times lower than conventional NPK ($p < 0.01$). Agronomic evaluation showed 19% higher maize shoot biomass ($p < 0.05$) and improved nitrogen use efficiency. Heavy metal concentrations (Pb: 38 ± 2 ; Cd: 0.9 ± 0.1 ; total Cr: $55 \pm 4 \text{ mg kg}^{-1}$) complied with EU 2019/1009 limits. This waste-derived composite offers a scalable, low-cost alternative to polymer-coated fertilizers, advancing circular economy principles in sustainable agriculture. Field-scale validation and Cr(VI) analysis are recommended for future work.

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