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Nitrogen, phosphorus and sulphur mineralization kinetic as affected by biochar, manure and ash amendments—An incubation study

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Abstract

Studies on the effect of biochar, manure and manure ash on soil nutrient kinetics are rare. Therefore, an incubation study was conducted to investigate nitrogen (N), phosphorus (P), and sulphur (S) kinetics in soils amended with biochar (B), B + cattle manure (BCM), B + goat and sheep manure (BGSM), B + poultry manure (BPM), farm yard manure (FYM), CM ash (CMA), GSMA, PMA, and wood ash (WA). Manure and manure ash were applied at 120 kg N ha⁻¹; B was applied at 5 tha⁻¹. Total mineral nitrogen (TMN), available P and S were determined at 2 weeks interval. Results revealed that soils treated with WA and PMA had the highest liming (pH 9.88) and salinity (820 μ S m⁻¹), respectively. The overall cumulative release of TMN, P and S in soil treated with PMA was 1, 7 and 16% higher than BPM amended soils, respectively. The least percentage of TMN, P and S mineralized was in soil treated with BCM (13.5%), BPM (12.3%) and WA (9.3%), respectively. The first order and power functions were able to capture the kinetic release of N, P and S in the soil and amendments. TMN had significant correlation with nitrate–N (r=0.925, $p \le 0.001$) ammonium-N (r=0.737, $p \le 0.01$), sulphate (r=0.823, $p \le 0.01$) and phosphate (r=0.702, $p \le 0.01$). The study concluded that the application of manure ash or manure with biochar encourages the timely release of TMN, P and S as well as improving the salinity and pH of the soil.

Keywords Ash · Biochar · C:N:P:S ratios · Kinetics · Nutrient release · Organic amendments

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Highlights

1. The application of wood ash or manure ash to the soil instead of biochar with manure consistently increased the pH and salinity of the soil.

2. The availability of phosphorus, sulphur, and total mineral nitrogen increased as a result of the conversion of manure to manure ash.

3. The release of TMN, available P, and S in the soil treated with biochar and manure-ash additions was captured by the first-order and power functions.

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Introduction

Animal manure has been reported as a rich source of plant nutrients, however, its application in the soil to supply nutrients such as nitrogen (N) (Roosta et al. 2016; Ashworth et al. 2020) can also lead to excessive application of phosphorus (P) (Rogeri et al. 2016; Bi et al. 2019; Bankole and Azeez 2024) and sulphur (S) (Hoang et al. 2020; Olawale et al. 2023; Bankole and Azeez 2024). Improper management of manure poses a serious challenge to the environment, agronomy and human health (Ashraf et al. 2021), such as odour emissions, greenhouse gas emissions such as methane and ammonia into the atmosphere, soil acidification, eutrophication (Zhang et al. 2020), leaching, and polluting underground water (Ding et al. 2016). An alternative method to mitigate the negative effect of animal manure on the environment is by converting it to manure ash (Song et al. 2020). The use of manure ash has been reported to be cost effective, attractive, and alternative source of chemical fertilizers that is free of pathogens and toxic substances, eliminates biological hazards (Tánczuk et al. 2019) and environmentally friendly (Codling 2006). The application of manure ash has been reported to increase mineralization and availability of nutrients due to the priming effects of the increased mineral input from soil organic materials (Doetterl et al. 2016) or decrease them due to the immobilization effect (Guo et al. 2018) due to the high amount of cations.

Another approach to minimizing nutrient losses and improving nutrient use efficiency is through the application of biochar (Ariyaratne 2000). Lehmann et al. (2011) defined biochar as the product of thermal degradation of organic materials in the absence of air (pyrolysis). As a soil amendment (Butnan et al. 2015), it acts as an adsorbent (Umeugochukwu 2016) and improves nutrient retention capacity (Laird et al. 2010). Biochar itself does not contain sufficient nutrients for plant growth (Yang et al. 2016), however, recent studies showed that biochar can adsorb nutrients (Ding et al. 2016) for it has a strong affinity for inorganic ions such as phosphate, sulphate, and nitrate (Kammann et al. 2015; Schmidt et al. 2015). It has been considered a potentially applicable material to mitigate nutrient loss due to its adsorption capacity (Gai et al. 2014).

Applications of biochar may be a crucial component for maintaining production while concurrently lowering pollution and fertilizer dependency, according to prior studies (Lehmann and Joseph 2015; Stavi and Lal 2013). More recently, numerous studies have shown that the addition of biochar to soils can increase crop yields and alleviate plant stresses associated with drought (Akhtar et al 2014), salinity (Dugdug et al. 2018), and heavy metals (Karunanayake et al. 2018; Rizwan et al. 2018). However, despite the increasingly diverse benefits of biochar application, impediments to the adoption of biochars in sustainable agriculture. Of concern is the great variability in biochars; not only in their nutrient availability and pH but also in their chemical (Gwenzi et al. 2014), and physical characteristics (Downie et al. 2009), depending on the nature of the feedstock and pyrolysis conditions (Barrow 2012).

Nitrate, phosphate, and sulphate are anions and may compete with each other for adsorption or colloidal sites, and their interactions may affect their availability, leading to nutrient loss if not properly managed (Brady and Weil 2010). Measuring the amount of nutrient released during a specified period under uniform conditions is an assessment of the mineralization potential of a soil or any organic material (Abbasi et al. 2003).

However, it remains to be established what mechanisms of interaction exist between biochar and manure for enhanced availability, long-term capture, and storage of nutrients in soils. Also, the availability of nutrients stored in soils as a function of biochar and manure application over time needs to be properly elucidated. Previous studies indicate that there has been very little or no retention of nutrients on biochars (Kameyama et al. 2016; Hollister et al. 2013; Gai et al. 2014).

Several approaches and methods have been used to evaluate mineralization kinetics as indicators of nutrient availability over time. Stanford and Smith (1972) proposed calculating the amount of N mineralized directly. Smith et al. (1980) improved the model and reported that a nonlinear least-squares equation gave a more accurate estimation of k and N₀. Azeez and Van Averbeke (2010) used the first order, second order, and power function models developed by Dang et al. (1994) for abiotic systems to estimate nitrogen mineralization kinetics in animal manures. It was reported that the model did not capture the N kinetics. Hence, there is a need to investigate the models used by Azeez and Van Averbeke (2010) on N, P, and S kinetics in soil treated with biochar, manure and manure ash. Lentz et al. (2014) highlighted the need to address the potential effects of biochar and manure ash on nutrient release in soils amended with various fertilizer sources by incubating under controlled conditions. However, scanty studies have been conducted to elucidate these. Therefore, this research was carried out to investigate the influence of biochar, manure, and animal manure ash on N, P, and S mineralization kinetics.

Materials and Methods

Soil samples collection, preparation and analyses

Soils were collected from fallow land within the depth of 0 to 20 cm at the Federal University of Agriculture Abeokuta (7° 14' 21" N, 3° 26' 89" E) with the aid of a shovel. The collected soil samples were air dried, pulverized, and composite samples passed through a 2 mm mesh sieve. The physicochemical properties were determined (Table 1). The prepared soil samples were analyzed for pH in a 1:2 soil-towater ratio using a glass electrode pH meter, and exchangeable acidity was determined by the titrimetric method according to McLean (1982). Soil electrical conductivity (EC) was measured in the suspension by the method of Jackson (1973). Particle size distribution was determined by the hydrometer method described by Bouyoucos (1951). Total organic carbon (TOC) was determined using the acid dichromate wet oxidation procedure of Nelson and Sommer (1996). Total nitrogen (TN) was determined by a modified micro-Kjeldahl digestion technique described by Jackson (1964). Nitrate and ammonium-nitrogen were determined spectrophotometrically (Cataldo et al. 1975). Available phosphorus (P) was extracted by Bray-1 procedure (Bray and Kurtz 1945) and determined colourimetrically. Available S was determined in 0.01 M CaCl2 extract (Williams and Steinbergs 1959) and was determined turbidimetrically by the procedure of Chesnin and Yien (1951). Exchangeable sodium (Na), potassium (K), calcium (Ca), and magnesium (Mg) were extracted with 1 N NH_4OAc buffered at pH 7.

Table 1 Properties of the experimental soil

Properties	Values
pH	6.15
Sand (g/kg)	892
Silt (g/kg)	48
Clay (g/kg)	60
Textural class	Sandy loam
EC (dS/m)	0.34
Organic carbon (g/kg)	6.49
Total nitrogen (g/kg)	0.43
Ammonium-N (mg/kg)	0.33
Nitrate–N (mg/kg)	6.89
Available phosphorus (mg/kg)	7.43
Available sulphur (mg/kg)	5.32
Calcium (cmol/kg)	4.21
Magnesium (cmol/kg)	2.04
Potassium (cmol/kg)	0.13
Sodium (cmol/kg)	0.23
Al+H (cmol/kg)	0.09
ECEC (cmol/kg)	6.70
Base Saturation (%)	98.66
Mn (mg/kg)	12.34
Fe (mg/kg)	23.45
Cu (mg/kg)	1.98
Zn (mg/kg)	8.89

The extracted Ca and Mg were determined using an Atomic Absorption Spectrophotometer (AAS) while extracted Na and K were determined using flame photometer. The effective cation exchange capacity (ECEC) was expressed as the sum of exchangeable cations and exchangeable acidity.

Manures Collection, Combustion and Analyses

Poultry, cattle, goat and sheep manures were collected from the College of Animal Science and Livestock Production Farm (FUNAAB). The management practices of these animals were semi-intensive except for poultry, which was intensive. Animal manures were stored as heaps in the animal units within the livestock building. The collected manures were air-dried and stored at room temperature. Poultry, cattle, goat and sheep manures were converted to manure ashes by combustion at a temperature of 600 °C in a closed furnace. Manure ashes were analyzed for nutrient composition by standard procedures (Kaira and Maynard 1991; Cater 1993).

Biochar Collection, Preparation and Analysis

Wood shavings were used for the production of biochar. The feedstock was collected from a commercial saw mill factory and air-dried before charring. The essence of air drying is to reduce the moisture content of the feedstock and hasten the charring process. Feedstock was weighed before and after charring so as to determine the biochar yield. Biochar yield is the proportion of the weight of the pyrolyzed product to the weight of the feedstock. The charring process will be carried out in a Top-lit-up-draft kiln (pyrolyzer). Feedstock was fed into the combustion chamber, and the temperature was set at 350 °C. Biochar produced was air dried, ground with a mortar and pestle to reduce their particle size, sieved to <2 mm, and homogenized prior to routine analysis, according to Kaira and Maynard (1991) and Cater (1993).

Laboratory Experiment

The incubation experiment was arranged in a completely randomized design with three replications. The treatments consisted of biochar, biochar + cattle manure (BCM), biochar + goat and sheep manure (BGSM), biochar + poultry manure (BPM), farmyard manure (FYM), cattle manure ash (CMA), goat and sheep manure ash (GSMA), poultry manure ash (PMA), wood ash (WA), and control (soil only). Soil samples were weighed at 300 g and transferred into 500 mL plastic containers. Manure ashes and manure were applied at 120 kg N ha⁻¹ according to Azeez and Averbeke (2010), while biochar was applied at 5 t ha^{-1} , and incorporated into the soil. The samples were covered with a perforated thin plastic layer for ventilation, moistened to 50% of the soil's moisture capacity. The temperature was regulated between 25 and 27 °C throughout the experiments. Samples were incubated in an incubator and analyzed fortnightly for 16 weeks for NO₃-N, NH₄-N, PO₄³⁻, SO₄²⁻, while total mineral N (TMN) was estimated as the sum of the NO₃-N and NH_4 –N.

Calculations

The percentage of total N, P, and S released from an applied source at time t was calculated as

$$\% \text{Mrelease} = \frac{Mt}{Mo} x 10 \tag{1}$$

Where M_t is TMN, P and S released at time t, M_0 is the total amount of N, P and S in manure sources, respectively. First order: (Qiu et al. 2009)

$$\ln Qt = \ln Qe - k_1 t \tag{2}$$

Second order: (Qiu et al. 2009)

$$1/Qt = 1/Qe + k_2t \tag{3}$$

Power function

Ot = atb

- first-order rate constant (day^{-1}) . K_1
- second-order rate constant (mg M kg⁻¹). K_2
- initial N release rate constant (mg M $kg^{-1} day^{-1}$). А
- release rate coefficient (mg M kg^{-1}). В

Qt (mg M kg⁻¹) is the amount of mineral M released after t days; Qe (mg M kg⁻¹) is the amount of mineral M released at equilibrium. M in the equations refers to mineral N, P, and S. Mineralization was assessed routinely by destructive sampling.

Statistical Analysis

Data were statistically analyzed by Analysis of Variance (ANOVA) using the GENSTAT statistical package (12th edition). Treatment means were separated for significant differences using Duncan's Multiple Range Test at 5% level of probability. The Microsoft Excel software package was used to make graphs. The correlation analysis coefficients between soil pH and EC, ammonium-N, nitrate-N, phosphate, and sulphate were analyzed using the Pearson nonparametric test.

Results

Soil and Amendments Characteristics

The textural class of the soil used for the study was sandy loam (Table 1). The pH of the soil was slightly acid (6.15) while the organic carbon content was low (6.49 g kg⁻¹). The soil was low in total nitrogen (0.43 g kg⁻¹), available P $(7.43 \text{ mg kg}^{-1})$, and S $(5.32 \text{ mg kg}^{-1})$, while the exchangeable cations of the soil were moderate, and their abundance was in this order: Ca > Mg > K > Na. The pH values of the amendments (Table 2) ranged from 13.18 in WA to 9.64 in BPM. The pH values of the biochar with or without manures were in the order of BGSM > BCM > B > BPM, while manure ashes were in the order of GSMA > PMA > CMA. The EC of the amendments ranged from 14.58 dS m^{-1} in WA to 3.77 dS m^{-1} in biochar.

Wood ash (WA) had significantly higher pH and EC values than other amendments; similarly, manure ashes had higher pH and EC than biochar mixed with manure. Biochar had significantly higher TOC compared to other amendments, however, there was no significant difference between the TOC of biochar mixed with manure and also between manure ashes; though biochar, BCM, BGSM, and BPM recorded significantly higher TOC than WA, CMA, GSMA, and PMA by 39.1, 13.6, 16.2, and 20.7%, respectively.

Biochar mixed with poultry manure had the highest total N, P, and S contents, followed by BCM, while biochar

Table 2 Characterization of amendments used for the study

BCM: Biochar+cattle manure; BGSM: Biochar+goat and sheep manure; BPM: Biochar+poultry manure; FYM: Farmyard manure; CMA: Cattle manure ash; GSMA: Goat and sheep manure ash; PMA: Poultry manure ash; WA: Wood ash; LSD: Least significant difference

Parameters	Biochar	BCM	BGSM	BPM	FYM	CMA	GSMA	PMA	WA	LSD
pН	10.66	10.21	10.89	9.64	11.64	11.26	12.05	11.81	13.18	1.52
$EC (dS m^{-1})$	3.77	4.08	4.87	7.64	8.85	10.37	11.41	11.94	14.58	4.23
Org. C (g/kg)	80.50	70.40	68.10	63.95	61.30	60.80	57.10	50.70	49.0	10.23
Total N (g/kg)	0.65	1.91	1.85	2.51	1.84	1.48	1.78	1.88	1.16	1.31
Total S (g/kg)	0.25	0.61	0.48	0.68	0.74	1.31	1.26	1.46	1.04	0.42
Total P (g/kg)	0.59	1.08	0.96	1.99	0.87	0.94	0.73	1.16	0.63	0.99
Calcium (g/kg)	0.83	3.73	3.87	4.06	1.03	3.67	2.75	2.67	6.42	1.84
Magnesium (g/kg)	0.27	1.85	1.84	2.87	0.21	0.48	1.47	1.43	3.49	0.67
Potassium (g/kg)	1.13	1.11	0.98	1.53	1.14	1.72	1.84	1.89	0.88	0.52
Sodium (g/kg)	0.51	0.39	0.38	0.51	0.27	0.93	0.89	1.21	0.37	1.81
C:S	384.00	228.5	237.50	216.5	97.01	54.00	53.01	41.02	55.02	122.60
C:P	157.90	101.35	103.15	87.1	82.10	75.40	91.5	51.10	90.60	45.77
C:N	123.80	36.96	36.91	25.53	34.06	41.08	32.08	26.97	42.24	8.57
Manganese (mg/kg)	34.10	195.52	174.63	287.71	670.84	1010.22	457.14	1471.71	38.71	523.10
Copper (mg/kg)	3.70	19.83	23.45	50.15	32.96	67.04	11.83	51.33	30.72	31.92
Iron (mg/kg)	121.90	1751.54	346.32	554.52	1246.71	2178.21	1780.41	2133.62	185.91	1215.50
Zinc (mg/kg)	9.83	314.53	273.14	409.54	476.53	2149.34	1457.12	2322.44	13.53	969.80

amendment had the least. Wood ash had the highest Ca and Mg contents, while the highest K and Na were recorded in PMA. Biochar recorded significantly higher C:N, C:P, and C:S while PMA had the least C:P and C:S, and BPM had the least C:N.

Effect of Amendments Mineralization on Soil pH and EC

The change in the soil pH with amendments over the incubation period is shown in Table 3. There was a significant increase in soil pH with the application of the amendments to the soil throughout the incubation period, except with BGSM and FYM at 10 and 14 weeks, respectively. Soil treated with WA recorded the highest pH values and was significantly higher than other amendments except CMA from 6 to 10 weeks of incubation. Soil treated with CMA, GSMA, and PMA consistently recorded higher pH values than BCM, BGSM, and BPM, respectively. In addition, the pH of soils amended with manure ash was significantly higher compared to soils treated with biochar mixed with or without manure and FYM from 10 to 16 weeks of the study. Soil pH consistently increased from 1 to 10 weeks in WA and CMA, and from 1 to 12 weeks in GSMA, while a continual decrease in soil pH was recorded in soils treated BCM and PMA throughout the entire incubation periods. The pH of soil treated with FYM slowly decreased from 4 to 16 weeks, while the pH of biochar amended soil was fairly consistent all through the study periods.

The EC in the control ranged from 77 to 120 μ S m⁻¹ and amendments had a significant increase in soil EC over the control (Table 3). Soil treated with WA had the highest EC for the first 2 weeks, while PMA amended soil recorded significantly higher EC compared to other amendments from 4 to 16 weeks, except soil treated with GSMA. All manure ash treatments (CMA, GSMA, and PMA) had significantly higher EC values than soils treated with biochar mixed with manures. However, BCM, BGSM, and BPM recorded significantly higher EC than biochar amended soil. A gradual increase in EC occurred as the weeks of incubation increased from 0 to 12 in soils treated with BPM, FYM, CMA, GSMA, and PMA, and decreased afterward until 16 weeks. Soils treated with BCD, BGSM, and WA were characterized by a gradual decrease in EC from 10 to 16 weeks of the incubation periods.

Treatments	1 Week	2 Week	4 Week	6 Week	8 Week	10 Week	12 Week	14 Week	16 Week
рН									
Soil	6.41	6.72	6.82	6.91	7.05	7.03	7.00	6.83	6.25
Biochar	7.81	7.88	7.84	7.86	7.86	7.85	7.83	7.84	7.80
BCM	8.69	8.62	8.54	8.41	8.25	7.92	7.50	7.43	7.23
BGSM	7.61	7.54	7.45	7.56	7.67	7.52	7.59	7.63	7.73
BPM	8.09	7.96	8.08	7.97	7.77	7.63	7.63	7.63	7.66
FYM	6.99	7.2	7.17	6.9	6.79	6.65	6.57	6.47	6.45
СМА	8.73	8.89	8.96	9.18	9.22	9.33	9.21	9.13	8.93
GSMA	8.34	8.46	8.48	8.51	8.58	8.67	8.71	8.63	8.57
PMA	8.99	8.97	8.92	8.73	8.41	8.36	8.25	8.17	8.12
WA	9.02	9.26	9.47	9.51	9.67	9.88	9.73	9.68	9.49
LSD	0.47	0.43	0.49	0.48	0.62	0.82	0.39	0.55	0.58
Electrical co	onductivity	$(\mu S m^{-1})$							
Treatments									
Soil	77	83	87	93	120	120	117	120	120
Biochar	143	180	273	343	340	290	267	253	247
BCM	253	373	410	450	437	450	420	363	307
BGSM	367	393	430	460	447	483	457	417	403
BPM	383	393	436	470	493	503	507	490	453
FYM	203	267	300	366	380	407	423	413	410
СМА	400	440	536	630	663	720	727	667	630
GSMA	410	483	557	643	693	737	760	730	713
PMA	427	483	610	690	760	800	820	760	740
WA	477	530	600	643	673	650	610	580	566
LSD	202.5	98.2	62.8	86.5	59.0	94.0	66.5	55.0	35.7

Table 3Effect of soil andamendments mineralizationon soil pH and electricalconductivity

Effect of Biochar, Manure and Manure Ashes on the Cumulative Release of a. Total Mineral N, Phosphate and Sulphate

The results of cumulative release of total mineral N (TMN) release in soil increased significantly ($p \le 0.05$) with the addition of manure ashes and biochar mixed with manures compared to control (unamended soil), biochar, and WA amended soils from 6 to 16 weeks of incubation (Fig. 1a). The cumulative release of TMN in soil treated with WA and biochar was not significantly different from the control.

Soil treated with FYM had the highest cumulative release of TMN, ranging from 42.4 to 474.9 mg kg⁻¹ followed by PMA (33.4 to 433.9 mg kg⁻¹) and GSMA (33.1 to 337.9 mg kg⁻¹) while the least was recorded in the control (16.2 to 143.99 mg kg⁻¹) followed by soil treated with wood ash (19.5 to 169.19 mg kg⁻¹) and biochar (21.4 to 204.79 mg kg⁻¹). The addition of manure ashes had a significantly higher TMN cumulative release than biochar and biochar mixed with manure, respectively. Soil treated with BPM, BGSM, and BCM had 52.4, 33.9, and 20.4% higher cumulative releases of TMN than biochar, respectively, while soil treated with PMA, GSMA, and CMA had 61.1,

49.9, and 47.8% higher cumulative releases of TMN than WA, respectively.

As shown in Fig. 1b, the application of manure ashes and biochar mixed with manure significantly increased the cumulative release of P compared to the control, biochar and WA treated soils. In general, soil treated with PMA had significantly the highest cumulative P release, ranging from 25.3 to 259.2 mg kg⁻¹ compared to other treatments, followed by BPM (19.2 to 245.2 mg kg⁻¹) while the control recorded the least (15.6 to 107.3 mg kg⁻¹). Soil treated with BPM, BGSM, and BCM had 51, 44.7, and 39.1% higher cumulative releases of P than biochar, respectively, while soil treated with PMA, GSMA, and CMA had 53.8, 38.3, and 47.4% higher cumulative releases of P than WA, respectively. The application of PMA and CMA recorded a significantly higher cumulative release of P than BPM and BCM from 2 to 16 weeks of incubation.

Soil treated with PMA had significantly ($p \le 0.05$) higher cumulative S (Fig. 1c) release at all the incubation periods than other treatments, ranging from 29 to 302.9 mg kg⁻¹, while the least was observed in the control, biochar, and WA treated soils, which recorded significantly lower cumulative S release compared to other treatments. The application of BPM recorded a significantly higher cumulative release of

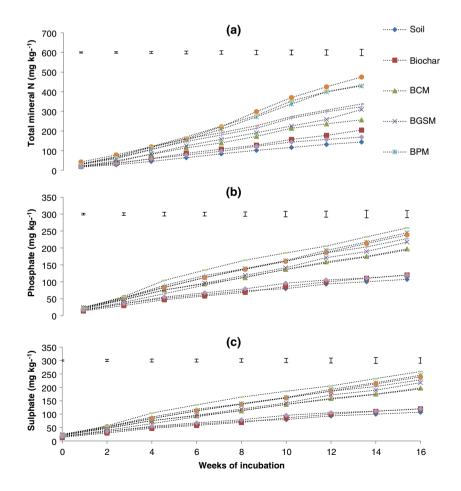


Fig. 1 Effect of biochar with manures and manure ashes on the cumulative release of (a) Total mineral N (b) Phosphate (c) Sulphate (bars=LSD bars)

S than other treatments except FYM. The overall cumulative release of S in soils treated with PMA and CMA was 14.1 and 27.6% higher than BPM and BCM amended soils, respectively, while BGSMA treated soil recorded a 3.6% higher cumulative release of S than GSMA.

Percentage of total mineral N, available P and S mineralized

Figure 2a shows soil treated with biochar recorded significantly higher TMN mineralization by 31.4%, followed by FYM (26.4%) and PMA (23.1%), while the least percentage of TMN mineralized was recorded in soil treated with BCM (13.5%), followed by BGSM (16.8%) and BPM (17.1%). Soils treated with BPM and BCM had significantly lower TMN mineralized compared to PMA and CMA, respectively. A significant high percentage (27.5%) of P mineralized from organic P (Fig. 2b) was recorded in soils amended with FYM, followed by GSMA (26.6%), compared to other treatments, while soil treated with BPM had a significantly lower percentage (12.3%) of P mineralized than other amendments. The percent of P mineralized in soils treated with BGSMA, GSMA, PMA, and CMA was significantly higher than soils amended with biochar and WA, respectively. The percentage of S mineralized (Fig. 2c) was higher in soils treated with BGSM (41.9%) and biochar (41.1%), followed by BPM (38.1%) and FYM (36%), while soil treated with WA had a significantly lower S mineralized compared to other amendments. However, soils treated with biochar mixed with manures had a significantly higher percentage of S mineralization than manure ashes.

Nitrogen, Phosphorus, and Sulphur Mineralization Kinetics

Table 4 shows the parameter estimates of the equation used to describe mineral N, P, and S kinetics in the control (soil only), soils treated with biochar mixed with manures and manure ashes. The coefficient of determination (\mathbb{R}^2) was used as the criterion for fit. Table 4 shows that the models were able to capture the mineralization pattern of mineral N in the amendments. The first and second-order constants

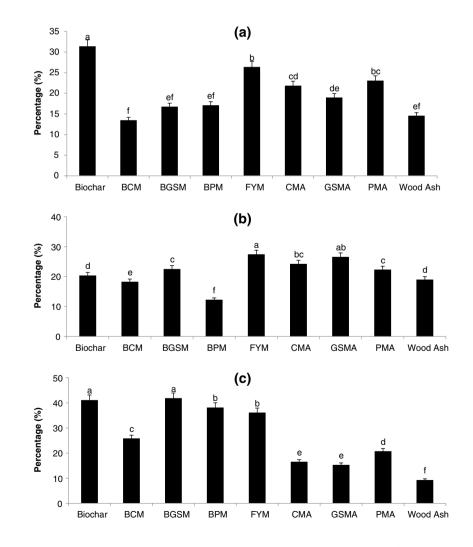


Fig. 2 Percentage (a) Total mineral N (b) Phosphate (c) Sulphate mineralized by biochar and ashed animal manures (bars = LSD bars)

 Table 4
 Estimated total mineral

 N, phosphate and sulphate
 kinetic models

First Order			Second Or	der	Power Function		
Estimated tota	al mineral N kin	netic models					
Treatment	K ₁	\mathbb{R}^2	K ₂	\mathbb{R}^2	а	b	\mathbb{R}^2
Soil	-0.130	0.92	-0.003	0.71	16.029	0.793	0.988
Biochar	-0.135	0.93	-0.002	0.73	20.381	0.813	0.997
BCM	-0.142	0.89	-0.002	0.62	23.536	0.875	0.995
BGSM	-0.159	0.88	-0.002	0.60	20.145	0.985	0.998
BPM	-0.161	0.93	-0.002	0.67	28.790	0.974	0.997
FYM	-0.147	0.95	-0.001	0.75	39.710	0.971	0.987
CMA	-0.135	0.89	-0.001	0.66	33.151	0.827	0.997
GSMA	-0.132	0.89	-0.001	0.64	36.199	0.812	0.996
PMA	-0.154	0.91	-0.001	0.64	32.579	0.841	0.997
WA	-0.128	0.90	-0.002	0.70	19.719	0.783	0.989
Estimated pho	sphate kinetic	models					
Soil	-0.102	0.83	-0.003	0.60	18.975	0.844	0.971
Biochar	-0.121	0.88	-0.003	0.63	15.529	0.746	0.989
BCM	-0.125	0.89	-0.002	0.61	23.466	0.773	0.986
BGSM	-0.124	0.91	-0.002	0.68	25.678	0.758	0.995
BPM	-0.138	0.86	-0.002	0.57	23.160	0.982	0.984
FYM	-0.13	0.87	-0.002	0.60	25.849	0.807	0.987
CMA	-0.121	0.86	-0.002	0.62	28.590	0.759	0.991
GSMA	-0.136	0.87	-0.003	0.60	19.244	0.948	0.992
PMA	-0.131	0.82	-0.002	0.66	29.733	0.832	0.976
Woodash	-0.108	0.84	-0.002	0.52	19.456	0.679	0.982
Estimated sulp	phate kinetic m	odels					
Soil	-0.143	0.92	-0.005	0.67	8.902	0.864	0.995
Biochar	-0.134	0.84	-0.005	0.56	10.349	0.941	0.977
BCD	-0.14	0.92	-0.003	0.68	15.021	0.846	0.993
BGSM	-0.139	0.91	-0.002	0.66	18.925	0.948	0.995
BPM	-0.139	0.90	-0.002	0.66	25.103	0.855	0.998
FYM	-0.155	0.89	-0.002	0.60	19.516	0.950	0.994
CMA	-0.135	0.89	-0.002	0.64	22.129	0.826	0.994
GSMA	-0.141	0.91	-0.003	0.66	17.861	0.859	0.996
PMA	-0.135	0.88	-0.002	0.63	32.068	0.819	0.995
Woodash	-0.102	0.93	-0.002	0.81	17.744	0.598	0.984

 $(K_1 \text{ and } K_2)$ were low and negative for all the amendments with the highest found in soil treated with BPM and the control, respectively. The first order's R^2 for all the amendments was higher than the second order though, soil treated with FYM recorded the highest R^2 in both orders. For the power function, soil treated with BGSM had the highest R^2 and coefficient (b); although the initial N release constant (a) was positive for all the amendments, the highest amount was observed in soil FYM, followed by GSMA, and the least in the control. The initial N release constant was observed to be higher in FYM and manure ashes than soil treated with biochar with or without manures.

The first and second-order constants for P and S mineralization were low and negative, respectively. In addition, the R^2 in the P and S kinetic models was higher in the first order compared to the second order in all the amendments; however, soil treated with BGSM had the highest R^2 in K_1 and K_2 for P mineralization, while soil treated with WA had the highest R^2 in K_1 and K_2 for S mineralization. For the power function model, the initial release rate constant (a) was highest in PMA for P and S, while the lowest was observed in the control. Additionally, the initial release rate in PMA and CMA was higher than that in BPM and BCM, respectively.

Relationship between soil nutrients, electrical conductivity and soil pH

The data on the correlation coefficient between soil pH, electrical conductivity, nitrate, ammonium, total mineral N, phosphate, and sulphate are presented in Table 5. From **Table 5** Relationship betweensoil nutrients, electricalconductivity and soil pH

	pН	EC	Ammonium-N	Nitrate-N	Total mineral N	Phosphate
EC	0.687*					
Ammonium-N	-0.08	0.266				
Nitrate-N	-0.077	0.431*	0.458*			
Total mineral N	-0.065	0.451*	0.737**	0.925***		
Phosphate	0.008	0.297	0.601*	0.622*	0.702**	
Sulphate	-0.028	0.445*	0.606*	0.791**	0.823**	0.738**

*, **, and *** significant at 5 and 1 and 0.1%, respectively

these results, it was indicated that electrical conductivity had a positive and significant relationship with soil pH ($r=0.687^*$), nitrate ($r=0.431^*$), TMN ($r=0.451^*$), and sulphate ($r=0.445^*$). There was a positive and significant relationship among all forms N with phosphate and sulphate, however, TMN had highly significant correlation with nitrate N ($r=0.925^{***}$) compared to ammonium-N ($r=0.737^{**}$). A positive and significant relationship between sulphate and phosphate ($r=0.738^{**}$) was observed.

Discussion

The soil properties indicated that the pH was slightly acidic which could be due to low exchangeable bases, especially in tropical regions. The considerable amount of accumulated litter and plant debris may have contributed to the relatively moderate amount of organic carbon content in the soil. The EC of the soil in its natural state was moderate; however, the total N, P, (Aghorunse et al. 2023) and S contents of the soil were low (Bankole et al. 2022) and slightly below the critical levels.

The high pH and EC values in soil amended with wood ash could be due to the presence of a considerable amount of neutralizing compounds and hydro-oxides of Ca, Mg, and K (Mandre et al. 2006) which are the main contributors to alkalinity in wood ash. Organic carbon is the main constituent of biochar, and in this order of abundance, BCM>BGSM>B PM>FYMA>CMA>GSMA>PMA, and the least was in wood ash (WA). Manure properties showed that manure from ruminant animals seems richer in organic carbon than nonruminant animals (poultry manure). The combined application of biochar with poultry manure (BPM) was richer in micronutrients, total N, S, and P than other amendments; this revealed that animal type, management system, diet or feed quality, additives such as growth stimulants and antibiotics, and the methods of manure storage could have accounted for higher nutrients in poultry manure compared to cattle, goat and sheep manures. Similar result was also reported by Bankole and Azeez (2024). The high carbon content of biochar materials was ascribed to the high C:N, C:P, and C:S ratios in biochar. In comparison to biochar, BCM, BGSM,

FYMA, CMA, GSMA, and wood ash, the high nutrient content and low C:N:P:S ratio in PMA and BPM may accelerate the breakdown and mineralization of manure and enhance the availability and release of N, P, and S. It's possible that the biochar's high C:N:P:S ratio was caused by the usage of wood shavings and high-temperature pyrolyzing at 350 °C.

Applying wood ash or manure ash to the soil consistently resulted in a considerable increase in pH values, which were greater than those of biochar with manure additives. Due to the high concentrations of Ca, Mg, and K, which are the main contributors to the soluble alkalinity in ash, the liming effect and neutralizing capability of ash were most likely the mechanism causing this rise (Etiegni and Campbell 1991).

The application of biochar is believed to improve soil pH; in this study, the pH of biochar was almost static and consistent, however, the combined application of biochar with manure raised the pH values. This may be attributed to ion exchange reactions that occur when the terminal OH⁻ of Al³⁺ and Fe²⁺ hydroxyl oxides are replaced by organic anions (Dikinya and Mufwanzala 2010) of the manure. Therefore, the ability of the combined application of biochar with manure to raise pH is due to the presence of exchangeable bases in the manure. Whalen et al. (2000) also reported that the increase in pH due to manure application was due to the buffering effect of cations in the manure. The highest pH was within 10 to 12 weeks of incubation in CMA, GSMA, and wood ash. In contrast, the combined application of biochar and manure had reater Ca, Mg, and K levels than the manure ashes and exhibited a more consistent, low, slow, and static liming effect throughout the incubation study. This could be attributed to the slow release of dissolved salts of Ca, Mg, K, and Na from the manures into the soil. This demonstrated that manure ash has a similar liming effect to wood ash. However, a sharp and gradual decrease in soil pH was characterized in manure ashes.

Nottidge et al. (2009) and Nweke (2018) have reported that exchangeable bases, effective cation exchange capacity (ECEC), and electrical conductivity (EC) increased in ash amended soil. All amendments significantly increased soil EC over the control; the increase in soil salinity was due to manure properties and the amount of basic cations. Soil salinization was initially greater in wood ash amended soil due to the readily available dissolved salts of Ca, Mg, K, and Na from the wood ash. Consequently, compared to other amendments, PMA, GSMA, and CMA had greater soil salt levels. The increased concentration of basic cations in the manure prior to burning, which led to a slow and continuous release of dissolved salts in the soil, may be the cause of the high EC in manure ashes.

Azeez and Averbeke (2010) reported that the increasing EC was a reflection of the amount of dissolved salts in the manures. This further buttressed the fact that liming and salinization effects are functions of manure quality and characteristics. Wood ash and manure ashes impacted a greater salinity effect than biochar, and the combined application of biochar and manure implies that the potential for soil salinization is higher in manure ashes, especially poultry manure ash. Moreover, it had been suggested that the release of base cations from wood ash and manure, sulphur mineralization, and nitrification of excess N were all factors in the rise in soil salinity. According to study, adding wood ash or biochar to organic manure may enhance microbial activity and nutrient release; this may be connected to the increase in salinity levels. The salinity of the soil is favorably impacted by the concentrations of nitrate, sulfate, and total mineral N. This might be explained by the possibility that the addition of salt from the amendments and the mineralization of manure, caused by the use of wood ash or biochar, increased the salinity of the soil.

The combined incorporation of biochar with manure or manure ashes resulted in higher total mineral N, sulphate, and phosphate compared to biochar and wood ash treated soil; this could be attributed to the high proportion of total N, P, and S in the manures before they were converted to manure ash. The incineration of manure to manure ash resulted in the greater availability of TMN, P, and S in their inorganic forms than the combined application of manure with biochar. Also, the differences in the fineness, surface area, and sizes between manure ash and manure with biochar played an important role in nutrient release and availability. Abbasi et al. (2007) reported that particle size plays a vital role in N mineralization as it affects the surface area of the N source and contact with microorganisms. Manure ashes also had a higher phosphate and sulfate cumulative release than the combined application of biochar and manure; PMA > BPM and CMA > BCM throughout the study. In addition, manure ashes had a higher TMN cumulative release than the combined application of biochar and manure, GSMA > BGSM, and PMA > BPM. The ability of the manure to mineralize and the nutrients released being adsorbed on the large surface area of biochar could be the reasons for the low release of TMN, phosphate, and sulfate in soil treated with a combined application of biochar and manure.

This had proven to be efficient at reducing dissolved soluble nutrients such as ammonium, nitrate, phosphate, and sulphate by adsorption; a similar finding was reported by Yao et al. (2012). A recent study showed that biochar, as an adsorbent (Umeugochukwu 2016), it has the ability to retain nutrients. Kammann et al. (2015) and Schmidt et al. (2015) also reported, that biochar has a strong affinity for inorganic ions such as phosphate, sulphate, and the total mineral N. Codling (2006) reported that the amount of P and S in the manure ash is similar to that in manure, while manure ash is more concentrated. This explains the rapid release of phosphate and sulphate in poultry and cattle manure ashes than in combined application of biochar with manure. Bauer et al. (2019) and Ervin (2019) observed that manure ash had similar efficiency to chemical fertilizers such as triple superphosphate and potassium sulphate.

However, the use of these chemical fertilizers further acidifies the soil. In the case of soils with relatively low pH, according to this study, the use of manure ashes may increase and maintain the soil pH to a desired level, likewise improving the release rate of soil nutrients. The significant and highly positive relationships between mineralized total mineral N (ammonium-N and nitrate-N) and sulphate and phosphate demonstrated the ability of manure or manure to increase N, P, and S availability. The rate of organic N, P, and S mineralization as well as the cumulative release that may be sustained in the soil over time at their application are important factors to consider when evaluating manure ash and biochar with manure nutrient release efficiency. Despite the role of soil microorganism activities in the manure, the proportion of organic N and P that mineralized was higher in manure ashes compared to biochar mixed with manure. The low TMN and phosphate mineralized in BCM, BGSM, and BPM could be attributed to the high C:N:P ratio and properties of biochar.

However, Xu et al. (2018) reported that the addition of biochar decreases microbial activity and invariably could also reduce the amount of TMN and P availability; a similar finding was reported by Chintala et al. (2014). Contrary to the findings observed in S mineralization, the study showed that the combined application of biochar and manure resulted in a high percentage of sulphate mineralization. The influence of biochar application with manure on the increase or decrease proportion of nutrients mineralized in sandy loam soil may not be well understood, however, several authors (Naeem et al. 2017; Motaghian et al. 2019; Song et al. 2019; Herrmann et al. 2019; Purakayastha et al. 2019) have reported an increase in soil nutrient availability with the application of biochar.

The application of kinetics in estimating potentially available and release rate coefficients of N in soils was first proposed by Stanford and Smith (1972). The estimates of mineralization and availability made by incubating biochar with manure or manure ash-amended soil under controlled conditions in this study showed the N, P, and S mineralization-immobilization dynamics of amended soil. The first and second order constants $(K_1 \text{ and } K_2)$ of the estimated total mineral N, phosphate, and sulphate kinetics were negative and low in all the amendments, however, the wide variation in the mineralization rate constant between biochar mixed with manure and manure ashes could be attributed to the properties of the manure and manure ashes. The initial TMN, phosphate, and sulphate release rate constants were positive for all the amendments and higher in manure ashes compared to the combined application of biochar with manure. This could be due to the lower C:N:P:S ratios of manure ashes, which release nutrients faster due to their inorganic form. Similar results have been reported by Olowoboko et al. (2019). The first order and power functions were able to capture the release of N, P, and S in the soil and amendments, since the R² used as the criterion of fit was high compared to the second order. The present findings obeyed the model proposed by Smith et al. (1980), but contrary to the findings of Azeez and Averbeke (2010) and Olowoboko et al. (2019) that the first order, second order, and power functions were unable to capture the release pattern of N due to the non-conformity of the data to the models, which may be ascribed to the inconsistent nutrient release patterns, mineralization, immobilization and fixation. Nitrogen, P, and S were readily mineralized in soil treated with either manure ashes or a combined application of biochar and manure; this can be attributed to the inorganic form of N, P, and S in manure ashes or organic forms in manure, which were later mineralized by soil microbes. The application of manure has been reported to reduce P fixation, (Azeez and Averbeke 2010) by replacing the sites that fix P with organic anions and increasing P, N (Wu et al. 2017) and S availability.

Conclusion

The findings from the study revealed that the application of manure ash or the combination of biochar and manure improved the chemical properties of the soil. Soils treated with manure ash had a greater liming and salinization effect in the incubation periods compared to the combined application of biochar with manure. The amendments quality and properties are the pronounced determinate of N, P, and S release. The fineness, larger surface area, and concentrated nutrient composition in the mineral form of manure ash improved and increased the cumulative release of N, P, and S and the percentage mineralization of N and P over the combined application of biochar with manure. From the incubation study, the second and power function exponential models fitted well with the observed N, P, and S mineralization data of the amended soil. Further research is needed to investigate the long-term impact of manure ashes and biochar powered by manure on the kinetic release of soil nutrients in cultivated soils. The study concludes that the application of biochar with poultry manure or poultry manure ash is a good liming material in acidic soils, improves soil salinity, and gradually increases the release of N, P, and S.

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Authors Contribution Dr. Bankole Ganiyu Olawale initiated the research, collected and analyzed data, and wrote the original draft. Dr. Aghorunse Adeoba assisted in proofreading and editing the manuscript. Prof. Jamiu Azeez supervised the research and reviewed the manuscript before submission. All authors have read and approved the final and revised version of the manuscript.

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Data Availability Data available at reasonable request.

Declarations

Conflict of Interest The authors certify do not have any conflicting interest or affiliation with any organization or entity with any financial or non-financial interest in the subject matter or material discussed in this manuscript.

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