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Responses of Extractable Phosphorus, Zinc and Related Soil Properties to Triple Superphosphate–Zinc Sulphate Combinations in an Acidic Tea-plantation Soil

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Authors' contributions

This work was carried out in collaboration among all authors. All authors contributed to the study design, investigation, data analysis and manuscript preparation. All authors read and approved the final manuscript.

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Abstract

Aims: To characterise extractable phosphorus and zinc pools and related properties in acidic tea-estate soil following incubation with nine combinations of triple superphosphate (TSP) and zinc sulphate.
Study Design: A completely randomised 3 × 3 treatment-combination experiment with three replicate soil containers per combination (27 experimental units).
Place and Duration: Surface soil from Birganj Tea Estate, Dinajpur, Bangladesh, was incubated for 90 days at 25 °C.

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Methodology: A sandy loam soil with initial pH 5.02 received TSP at 40, 80 or 120 kg product ha^{-1} together with $ZnSO_4 \cdot 7H_2O$ at 5, 10 or 20 kg product ha^{-1}. Post-incubation measurements comprised pH, electrical conductivity (EC), organic matter, total nitrogen, exchangeable potassium, available sulfur, Olsen-extractable phosphorus and DTPA-extractable zinc.
Results: Olsen-extractable phosphorus ranged from 11.32 to 32.25 mg kg^{-1} and was highest in treatments T8 and T9, both receiving 120 kg TSP ha^{-1}. DTPA-extractable zinc ranged from 1.45 to 3.13 mg kg^{-1}; within each TSP group, the highest zinc means occurred at 20 kg $ZnSO_4$ ha^{-1}. Soil pH was lower under the higher-input combinations, whereas EC, exchangeable potassium, organic matter and available sulfur exhibited distinct treatment patterns. Total nitrogen varied within a narrow interval.
Conclusion: The combinations yielded differentiated post-incubation phosphorus and zinc extraction profiles in a single acidic tea-plantation soil. The results identify treatment patterns for evaluation in planted-soil and field experiments.

Keywords: Acidic tea soil; Camellia sinensis; DTPA-extractable zinc; Olsen-extractable phosphorus; phosphorus–zinc nutrition; soil incubation.

1. Introduction

Tea (*Camellia sinensis* L.) cultivation in Bangladesh is concentrated in the northeast and southeast, with an expanding production area in northern districts including Panchagarh, Thakurgaon and Dinajpur. The suitability of northern soils for tea reflects several favourable physical characteristics, although acidity and heterogeneous nutrient status present practical challenges for fertiliser management (Uddin *et al*., 2020). Local land-use change has also been associated with differences in soil organic matter and fertility properties around Birganj (Khan & Alam, 2025). Soil-specific characterization is therefore important when evaluating the nutrient response to fertiliser inputs in this production region. A comprehensive meta-analysis of Chinese tea plantations similarly demonstrated that soil nutrient status and acidity vary with geographical location, soil characteristics and fertiliser management, reinforcing the importance of site-specific nutrient assessment (Wang *et al*., 2025).

Phosphorus (P) and zinc (Zn) are essential for tea growth, but their behaviour in acidic soil depends on the soil matrix and the chemical environment of the root zone. Phosphate can react with iron- and aluminium-bearing mineral surfaces, while the extractability of Zn depends on pH, mineral sorption, organic ligands and competing ions. Physiological studies of tea have demonstrated that Zn stress is associated with changes in photosynthesis, growth and biochemical processes (Mukhopadhyay *et al*., 2013). Investigations of tea-plantation soils further link soil acidification with changes in P fractions and the distribution of extractable micronutrients (Lin *et al*., 2025; Ye *et al*., 2025).

The interaction between P and Zn varies with soil composition, fertiliser supply and plant uptake. In wheat, high P supply can lower tissue Zn under particular soil conditions, whereas other experiments identify P–Zn responses that depend strongly on the soil and the rates used (Ova *et al*., 2015; He *et al*., 2021; Nath *et al*., 2024). Plant regulation also influences how nutrient additions translate into shoot and grain concentrations (Nadeem *et al*., 2024). A field investigation in northern Bangladesh evaluated soil and foliar Zn fertilisation in tea (Khan *et al*., 2025), but extractable P–Zn profiles under controlled incubation were not its primary outcome. A plant-free incubation provides a complementary chemical perspective by examining the extractable nutrient pools remaining after a defined contact period.

This study examined a composite acidic soil from Birganj Tea Estate incubated with three TSP product rates crossed with three zinc sulphate product rates. The principal objectives were to compare post-incubation Olsen-extractable P and DTPA-extractable Zn across the nine combinations, characterise associated responses in soil pH, EC, organic matter, total N, exchangeable K and available S, and describe associations among these measured properties. The working expectation was that higher TSP and zinc sulphate inputs would be reflected in differentiated extractable P and Zn pools, with associated changes in other soil-test properties.

2. Materials and Methods

2.1 Soil Collection and Initial Properties

Surface soil (0–15 cm) was collected from Birganj Tea Estate, Birganj, Dinajpur, Bangladesh (25.872854° N, 88.628326° E). The location is within Agro-Ecological Zone 1. The soil was described as a non-calcareous brown floodplain soil of the Ranisankail series. Field subsamples were composited, transported to the laboratory, air-dried, gently crushed and passed through a 2-mm sieve. The composite contained 58% sand, 32% silt and 10% clay, with a measured water-holding capacity of 48.84%. Initial soil properties were pH 5.02, organic matter 0.84%, total N 0.15%, Olsen-extractable P 8.00 mg kg^{-1}, exchangeable K 1.26 $cmol_c$ kg^{-1}, available S 26.27 mg kg^{-1} and DTPA-extractable Zn 1.56 mg kg^{-1}.

2.2 Treatment Structure and Incubation

The incubation used a 3 × 3 treatment arrangement in a completely randomised design with three replicate containers per combination (27 experimental units). The applied TSP product rates were 40, 80 and 120 kg ha^{-1}, and the zinc source was zinc sulphate heptahydrate ($ZnSO_4 \cdot 7H_2O$) at product rates of 5, 10 and 20 kg ha^{-1}. The stated rates refer to fertiliser products rather than elemental P, elemental Zn or P_2O_5. Table 1 identifies the nine combinations; each TSP level was paired with every zinc sulphate level.

Each plastic container received 1 kg of air-dried soil. Fertiliser quantities corresponding to the nominal field application rates were converted to the soil mass and thoroughly mixed with each aliquot. Containers were periodically weighed, and distilled water was added to maintain moisture close to field capacity. Perforated lids permitted gas exchange while limiting evaporative loss. Samples were incubated in darkness at 25 °C for 90 days and then air-dried, crushed and passed through a 2-mm sieve before laboratory measurements.

Table 1. Treatment combinations in the 3 × 3 soil-incubation experiment

Treatment	TSP product rate (kg ha^{-1})	$ZnSO_4 \cdot 7H_2O$ product rate (kg ha^{-1})
T1	40	5
T2	40	10
T3	40	20
T4	80	5
T5	80	10
T6	80	20
T7	120	5
T8	120	10
T9	120	20

2.3 Soil Chemical Analyses

Soil pH was measured using a glass-electrode meter at a 1:1.25 soil-to-water ratio. Electrical conductivity was determined in a 1:5 soil-to-water extract. Organic carbon was measured by wet oxidation and multiplied by 1.73 to express organic matter (Walkley & Black, 1934). Total N was measured by the micro-Kjeldahl method (Bremner & Mulvaney, 1982). Phosphorus was extracted with 0.5 M $NaHCO_3$ following Olsen *et al*. (1954) and quantified colourimetrically using a UV–Vis spectrophotometer (UV-1900, Shimadzu, Kyoto, Japan). Exchangeable K was determined by flame photometry. Available sulfur was determined turbidimetrically after $BaCl_2$ precipitation using the UV–Vis instrument. Zinc was extracted with diethylenetriaminepentaacetic acid (DTPA) following the soil-test approach of Lindsay and Norvell (1978) and measured by atomic absorption spectrophotometry (AA-7000, Shimadzu, Kyoto, Japan).

The P and Zn values are designated as Olsen-extractable P and DTPA-extractable Zn throughout. These extractant-defined measurements describe operational nutrient pools under specified laboratory conditions, rather than directly calibrated tea uptake or crop response. The analytical properties, extraction approaches and reporting units are summarised in Table 2.

Table 2. Soil analytical methods and reporting units

Soil property	Method or extractant	Unit
pH	Glass electrode; soil:water 1:1.25	—
Electrical conductivity	Conductivity meter; soil:water 1:5	dS m^{-1}
Organic carbon	Wet oxidation	%
Organic matter	Organic C × 1.73	%
Total nitrogen	Micro-Kjeldahl digestion and distillation	%
Olsen-extractable P	0.5 M $NaHCO_3$; UV–Vis colorimetry (UV-1900)	mg kg^{-1}
Exchangeable K	Flame photometry	$cmol_c$ kg^{-1}
Available sulfur	$BaCl_2$ turbidimetry; UV–Vis (UV-1900)	mg kg^{-1}
DTPA-extractable Zn	DTPA extraction; atomic absorption (AA-7000)	mg kg^{-1}

2.4 Statistical Analysis

Results are expressed as treatment means ± standard error (n = 3). The nine fertiliser combinations were compared as a single treatment factor using Statistix 10. Fisher's least significant difference procedure at the 5% probability level was used for reported treatment-mean groupings. The analysis describes differences among the nine combinations rather than separately estimating TSP and $ZnSO_4$ main effects or their interaction. Pearson correlation coefficients among post-incubation soil measurements were summarised descriptively.

3. Results and Discussion

3.1 Soil pH and Electrical Conductivity

Soil pH differed across the nine fertiliser combinations after 90 days (Fig. 1a). The highest mean was observed in T1 (40 kg TSP and 5 kg $ZnSO_4$ ha^{-1}), while T9 (120 kg TSP and 20 kg $ZnSO_4$ ha^{-1}) was among the lowest. The broad tendency toward lower pH in several higher-input combinations is consistent with the sensitivity of acidic tea soils to nutrient inputs, although fertiliser composition, soil buffering and reaction time influence the direction and extent of such changes. The importance of soil reaction for tea nutrition was further demonstrated by Jia *et al*. (2024), who reported that variation in soil pH influenced the uptake and enrichment of several essential elements in tea plants. Long-term changes in tea-garden soil chemistry under fertilisation have also been documented in Bangladesh (Jahan *et al*., 2022).

Electrical conductivity measured in the 1:5 soil–water extract remained numerically low across treatments but varied in its treatment pattern (Fig. 1b). T3 had the lowest plotted mean, whereas T7 and T8 were among the highest. These observations summarise water-extractable salt concentrations after incubation and should be interpreted using the stated extraction ratio. Taken together, Fig. 1 shows that the treatment combinations altered the measured soil reaction and soluble-ion environment without establishing a single causal chemical pathway.

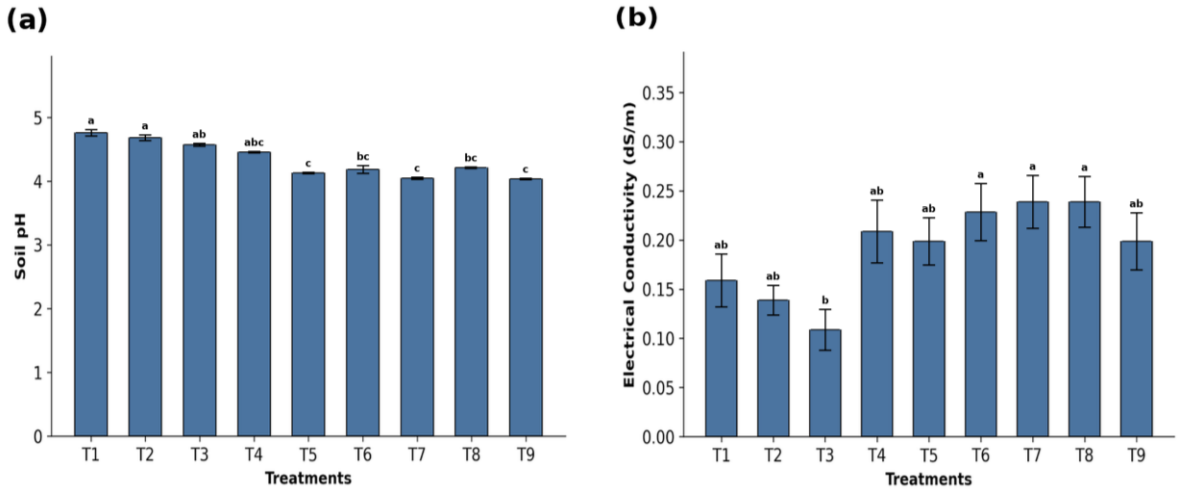


Fig. 1. Soil pH (a) and electrical conductivity in a 1:5 soil–water extract (b) following 90 days of incubation. Bars indicate treatment means ± standard error (n = 3). Lowercase letters denote the treatment groupings from the reported Fisher least significant difference analysis at the 5% probability level

3.2 Organic Matter and Total Nitrogen

Post-incubation soil organic matter ranged from approximately 0.57% to 0.80%, compared with 0.84% measured in the initial composite soil (Fig. 2a). The lowest mean occurred in T9, whereas most other combinations clustered in a narrower, higher interval. Total N ranged from 0.11% to 0.14% and the reported treatment means belonged to the same statistical group (Fig. 2b). Neither fertiliser source supplied a nitrogen fertiliser or organic amendment, so the results primarily characterise the background organic matter and bulk nitrogen pools present in the incubated samples.

Changes in extractable nutrients may occur while total soil N remains relatively constant, because total N integrates chemically diverse pools that can respond slowly over a 90-day period. Regional surveys have documented differences in soil organic matter associated with land-use history in the Dinajpur area (Khan & Alam, 2025). Fig. 2 provides useful context for interpreting the more pronounced P and Zn extraction responses against the relatively narrow variation in organic matter and total nitrogen.

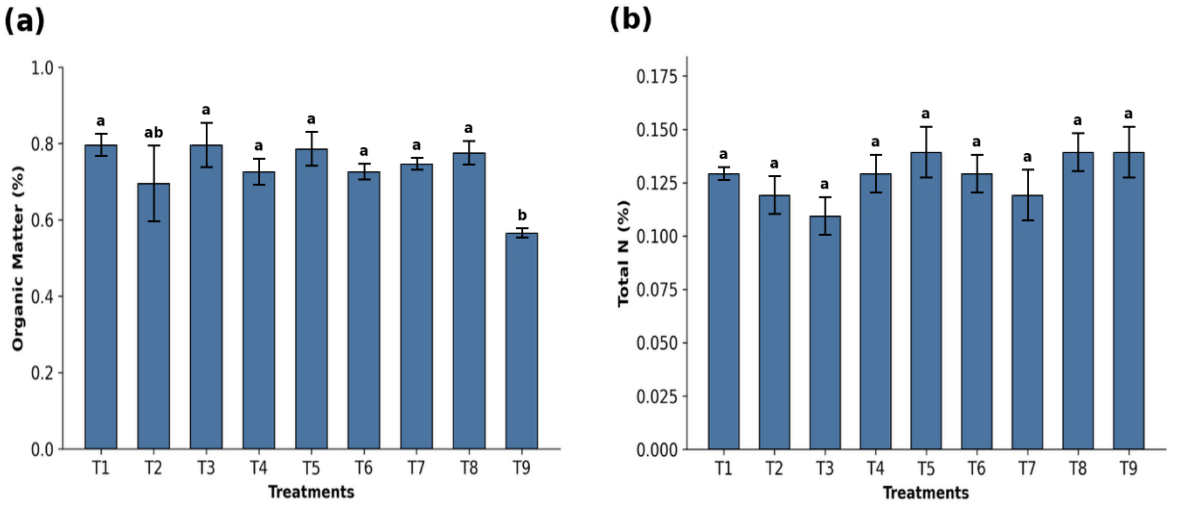


Fig. 2. Soil organic matter (a) and total nitrogen (b) after 90 days of incubation. Bars indicate treatment means ± standard error (n = 3). Lowercase letters denote the treatment groupings from the reported Fisher least significant difference analysis at the 5% probability level

3.3 Exchangeable Potassium and Available Sulfur

Exchangeable potassium varied within a relatively narrow range of 1.20–1.38 $cmol_c\ kg^{-1}$ across the nine combinations (Fig. 3a). The largest plotted mean occurred in T9, while T1 and T6 were among the lower means. Potassium was not supplied by either of the two fertiliser products, so these values primarily describe the exchangeable pool recovered from the incubated soil matrix.

Available sulfur exhibited a wider distribution, from 11.34 to 35.68 mg kg^{-1} (Fig. 3b). T4, combining 80 kg TSP ha^{-1} with 5 kg zinc sulphate ha^{-1}, showed the largest mean, whereas T1 showed the lowest. Unlike the exchangeable-K response, sulfur concentrations did not increase consistently with the nominal zinc sulphate rate. Because $ZnSO_4 \cdot 7H_2O$ supplied both Zn and sulfate, the observed sulfur pattern reflects the combined consequences of the supplied material and its extractability after incubation. Fig. 3 illustrates these contrasting responses of a nutrient not present in the fertiliser inputs (K) and a nutrient co-supplied with zinc (S).

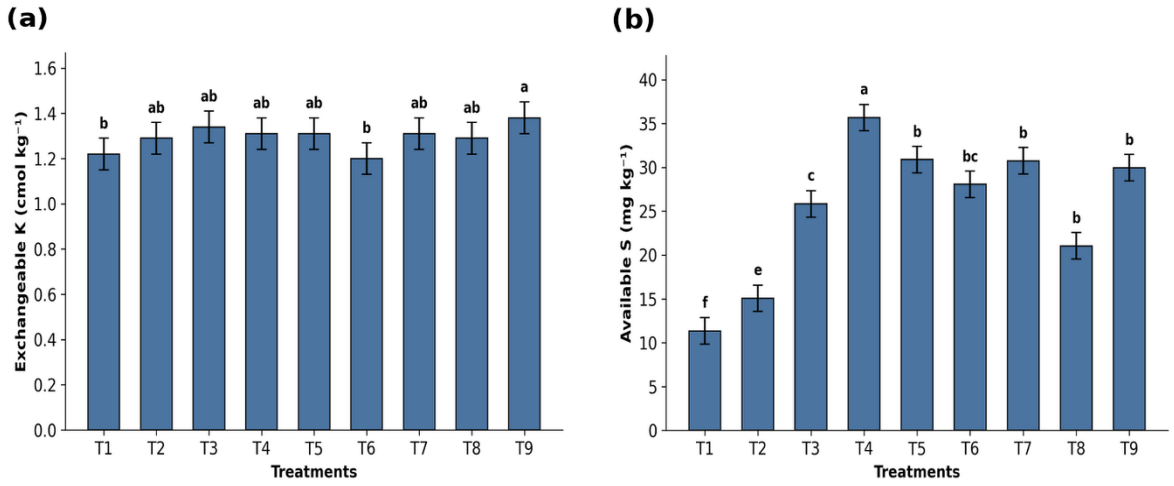


Fig. 3. Exchangeable potassium (a) and available sulfur (b) after 90 days of incubation. Bars indicate treatment means ± standard error (n = 3). Lowercase letters denote the treatment groupings from the reported Fisher least significant difference analysis at the 5% probability level

3.4 Olsen-Extractable Phosphorus

Olsen-extractable phosphorus ranged from 11.32 mg kg^{-1} in T1 to 32.25 mg kg^{-1} in T8 (Fig. 4a). Within the group receiving 40 kg TSP ha^{-1}, mean Olsen-P increased from T1 through T3 as zinc sulphate rates rose. The corresponding within-group patterns at 80 and 120 kg TSP ha^{-1} were less uniform. The largest means occurred in T8 and T9, both of which received 120 kg TSP ha^{-1}. The nine-combination comparison therefore identified a higher post-incubation bicarbonate-extractable P pool in two of the combinations with the largest TSP product input.

The magnitude and direction of P–Zn responses depend on the soil and biological context. Bibi *et al*. (2020) reported effects of P and Zn fertiliser rates and timing on wheat grain nutrient outcomes, whereas He *et al*. (2021) documented strong P–Zn interactions in a calcareous soil–alfalfa system. Their findings establish that interactions are context-dependent rather than universally antagonistic. Studies in planted tea systems likewise demonstrate the role of rhizosphere processes: phosphorus-solubilizing bacteria altered nutrient profiles of tea rhizospheres (Guo *et al*., 2024), and long-term organic P substitution was linked to microbial restructuring and P bioavailability in acidic tea soils (Jiang *et al*., 2026).

Bicarbonate extraction quantifies a defined operational P pool, not the total phosphate content or the quantity necessarily absorbed by tea plants. Work on tea plantations has linked acidification with shifts in labile and less-labile phosphorus fractions (Lin *et al*., 2025). A regional investigation of tea plantations in Yunnan also identified substantial spatial variation in soil available phosphorus, with soil chemical properties emerging as major determinants of its distribution (Long *et al*., 2026). In this incubation, the treatment pattern is most directly interpreted as a response in Olsen-extractable P after 90 days. The paired comparison with zinc is presented in Fig. 4, which also identifies the DTPA-extractable Zn pool for each combination.

3.5 DTPA-Extractable Zinc and the P–Zn Relationship

DTPA-extractable zinc ranged from 1.45 to 3.13 mg kg^{-1} (Fig. 4b). At each TSP rate, the largest mean occurred with the highest zinc sulphate product rate (20 kg ha^{-1}), represented by T3, T6 and T9. At 40 kg TSP ha^{-1}, the mean rose from 1.52 mg kg^{-1} in T1 to 2.73 mg kg^{-1} in T3. The same ordering of the highest $ZnSO_4$ input was evident within the 80 and 120 kg TSP groups. This recurring within-group pattern provides a clear description of the Zn extraction response to the nine fertiliser combinations.

Soil DTPA-Zn and plant Zn uptake are related but distinct endpoints. Plant studies have shown that phosphorus supply can influence tissue Zn through several pathways, with responses differing among native soil, sterilized soil and nutrient solutions

(Ova *et al*., 2015). Dhaliwal *et al*. (2023) reported that P and Zn management outcomes in wheat depended on the initial soil P status, and Khan *et al*. (2025) demonstrated that soil and foliar Zn application methods influenced tea yield in northern Bangladesh. Those findings complement the laboratory evidence without establishing a yield response for the incubated soil. In a long-term fertilization and cropping study in Bangladesh, treatments receiving phosphorus as triple superphosphate had higher extractable and soil-solution Zn concentrations (Khan et al., 2002). Differences in fertilization history and cropping conditions limit direct comparison with the present laboratory incubation.

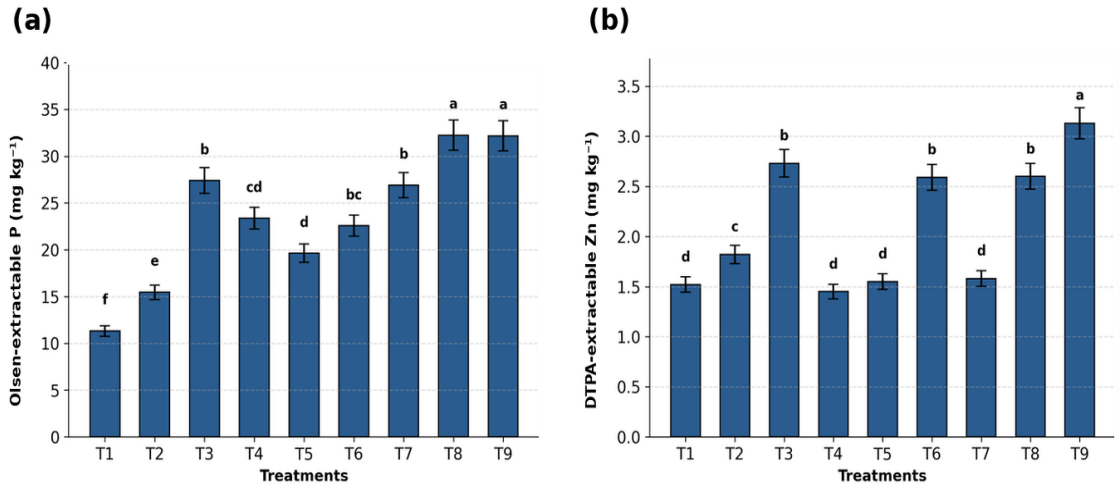


Fig. 4. Olsen-extractable phosphorus (a) and DTPA-extractable zinc (b) after 90 days of incubation. Bars indicate treatment means ± standard error (n = 3). Lowercase letters denote the treatment groupings from the reported Fisher least significant difference analysis at the 5% probability level

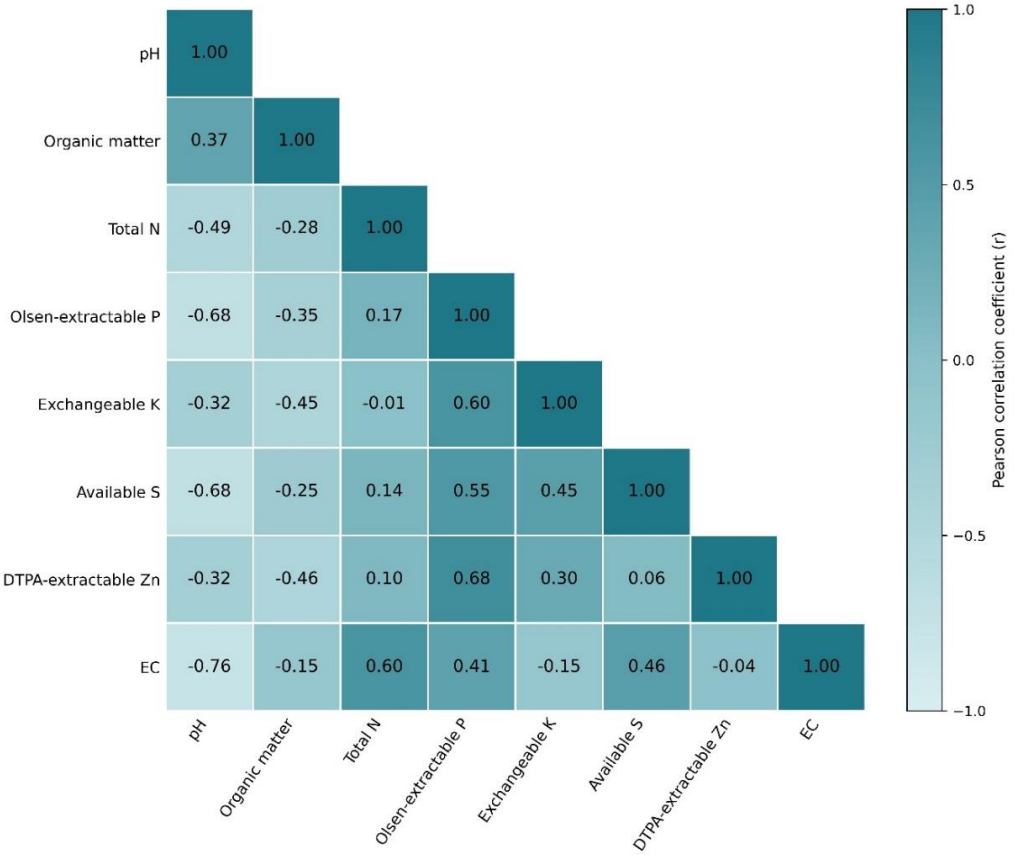


Fig. 5. Pearson correlation coefficients among post-incubation soil properties. P, Olsen-extractable phosphorus; K, exchangeable potassium; S, available sulfur; Zn, DTPA-extractable zinc; EC, electrical conductivity. Coefficients summarize associations and do not imply causation

The DTPA method measures an extractant-defined pool that includes zinc associated with several soil binding environments. In strongly acidic tea soils of northern Vietnam, Hoang *et al*. (2026) observed positive associations between DTPA-extractable zinc and several relatively labile zinc fractions, including exchangeable and organic matter-bound pools. Regional tea-garden soil measurements from Assam also illustrate that baseline DTPA-Zn values differ among sites (Borah *et al*., 2024). Fig. 4 jointly summarizes the principal result of the present experiment: The P extraction profile was greatest in T8/T9, whereas Zn extraction was greatest at the highest zinc sulphate rate within each TSP group.

3.6 Associations Among Measured Soil Properties

The correlation matrix presents descriptive relationships among the post-incubation soil properties (Fig. 5). Soil pH was negatively correlated with EC ($r = -0.76$), Olsen-extractable P ($r = -0.68$), available S ($r = -0.68$) and total N ($r = -0.49$), and positively correlated with organic matter ($r = 0.37$). Olsen-extractable P was positively correlated with DTPA-extractable Zn ($r = 0.68$), exchangeable K ($r = 0.60$) and available S ($r = 0.55$). Organic matter showed negative correlations with DTPA-Zn ($r = -0.46$), exchangeable K ($r = -0.45$) and total N ($r = -0.28$).

These associations integrate the covariation of measured properties across treatment conditions. The positive Olsen-P–DTPA-Zn correlation is compatible with overlapping trends in extractable nutrient pools but does not demonstrate that one fertiliser directly mobilized the other nutrient. Likewise, the inverse pH relationships with P and S describe the observed soil chemistry without independently establishing an acidification mechanism. Similar links among soil acidity, phosphorus and micronutrient measures have been investigated in regional tea-soil surveys (Ye *et al*., 2025). Fig. 5 summarizes the direction and magnitude of the descriptive associations.

4. Conclusion

A 90-day incubation of acidic tea-estate soil with nine TSP–zinc sulphate combinations produced differentiated extractable phosphorus, zinc and sulfur profiles. Olsen-extractable P ranged from 11.32 to 32.25 mg kg^{-1} and reached its largest means in T8 and T9, the combinations containing 120 kg TSP ha^{-1}. DTPA-extractable Zn ranged from 1.45 to 3.13 mg kg^{-1}; within each TSP group, the highest Zn mean occurred with 20 kg zinc sulphate ha^{-1}. Exchangeable K and total N varied over narrower intervals, while soil reaction, EC and organic matter displayed their own treatment patterns. These findings provide a site-specific chemical baseline for subsequent studies linking soil P–Zn extraction behaviour to nutrient uptake and tea performance.

5. Limitations of the Study

The study characterised one composite tea-estate soil after a single 90-day plant-free incubation, so its findings concern laboratory soil-test pools rather than crop uptake, tea yield or wider regional soil responses. No contemporaneously incubated zero-TSP or zero-$ZnSO_4$ controls were included. The analysis compared treatment combinations as a single factor, and the Pearson coefficients are descriptive. Olsen-P and DTPA-Zn were measured as method-defined indices without a tea-specific calibration. Exact per-container fertiliser quantities, individual replicate observations and complete inferential output are required for independent quantitative validation; additional planted-soil experiments would establish the agronomic relevance of the observed nutrient patterns.

Data Availability

Data supporting the findings are available from the corresponding author on reasonable request.

Acknowledgements

OpenAI ChatGPT (OpenAI, web application; GPT-series model, accessed September–October 2026) assisted with language editing, manuscript organization and consistency checks. The authors evaluated and revised all suggested changes and accept full responsibility for the data, interpretation, originality and references.

Declaration of AI Use

This manuscript was prepared through the intellectual contributions of the author(s) to the study design, data analysis, interpretation of results, and scholarly content. ChatGPT, Claude and Perplexity were used solely to assist with grammatical refinement and reference formatting during manuscript revision. The author(s) accept full responsibility for the accuracy, integrity, and final content of the manuscript.

Competing Interests

Authors have declared that no competing interests exist.

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