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Fractionation and bioavailability of zinc in strongly acidic tea soils of northern Vietnam

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Abstract

Zinc (Zn) is an essential micronutrient for tea (*Camellia sinensis*) quality and productivity, yet its bioavailability in strongly acidic tea soils remains insufficiently understood. This study investigated the distribution of Zn fractions and their relationships with soil properties in tea garden soils of Thai Nguyen province, northern Vietnam. Zinc fractionation was determined using a sequential extraction procedure, and potentially available Zn was assessed by diethylene triamine pentaacetic acid (DTPA) extraction across different land uses (short-term tea cultivation, long-term tea cultivation, and adjacent forest soils) and soil types. Residual Zn (Res-Zn) constituted the dominant pool in all soils, followed by Fe oxide-bound Zn (FeO-Zn), organic matter-bound Zn (OM-Zn), acid-soluble Zn (Aci-Zn), Mn oxide-bound Zn (MnO-Zn), and exchangeable Zn (Ex-Zn). Statistical comparisons revealed that, among all Zn fractions, only FeO-Zn exhibited significant differences across tea cultivation duration, soil types, and land uses ($p < 0.05$), with significantly lower FeO-Zn contents in long-term tea plantations compared with short-term plantations and forest soils, indicating a cumulative effect of prolonged tea cultivation on Zn associated with Fe oxides under acidic conditions. Correlation analysis further showed that FeO-Zn was significantly related to soil pH and indicators of oxide crystallinity, whereas OM-Zn was positively associated with organic carbon content and cation exchange capacity. In addition, DTPA-Zn showed strong positive correlations with Ex-Zn, Aci-Zn, MnO-Zn, and OM-Zn, confirming that these relatively labile fractions contribute most to Zn bioavailability. Comparison with Mn fractionation in the same soils indicated contrasting controlling mechanisms, whereby Zn availability was primarily regulated by sorption-desorption processes and organic complexation, while Mn dynamics were more strongly influenced by oxide associations and redox-related transformations.

1. Introduction

Zinc (Zn) is an essential micronutrient required for the growth, development, and reproduction of plants, animals, and humans (Alloway, 2008). In plants, Zn plays a critical role in membrane integrity, protein synthesis, enzyme activation, and gene expression, and its deficiency commonly results in reduced crop yield and quality (Figueiredo et al., 2012; Sadeghzadeh, 2013). Although total Zn contents in agricultural soils typically range from 10 to 300 mg kg^{-1} (Kiekens, 1995), total Zn content alone provides limited information on its bioavailability, as Zn mobility, reactivity, and uptake by plants are strongly governed by its chemical fractions or speciation in soil (Li et al., 2003).

For tea (*Camellia sinensis* (L.) O. Kuntze), Zn is an essential micronutrient closely linked to both yield and quality, as adequate Zn supply enhances the accumulation of key bioactive compounds such as (–)-epigallocatechin gallate (EGCG) and flavan-3-ols (Herman et al., 2022), whereas Zn deficiency directly constrains plantation productivity and tea quality (Nelson, 2006; Huang et al., 2021; Khan et al., 2025). Tan Cuong commune in Thai Nguyen province, northern Vietnam, is a well-known high-quality tea-producing area; however, prolonged tea cultivation combined with intensive nitrogen fertilization has led to pronounced soil acidification, with soil pH frequently declining below 4.0 (Huu Chien et al., 2019). Such strongly acidic conditions accelerate nutrient leaching, modify mineral stability, and substantially influence the transformation, mobility, and bioavailability of trace elements, including Zn (Haynes and Swift, 1986).

Understanding Zn availability in soils, therefore, requires detailed knowledge of its distribution among specific chemical fractions. Sequential extraction approaches commonly differentiate Zn into exchangeable (Ex-Zn), acid-soluble (Aci-Zn), Mn oxide-bound (MnO-Zn), organic matter-bound (OM-Zn), Fe oxide-bound (FeO-Zn), and residual (Res-Zn) pools. Among these fractions, the exchangeable, acid-soluble, and organic matter-bound forms are generally considered the most labile and are most closely associated with plant uptake, whereas the residual fraction represents the most stable and least bioavailable pool (Singh et al., 2019). In parallel, diethylene triamine pentaacetic acid (DTPA) extraction is widely used to estimate potentially available Zn, reflecting the fraction of Zn that is readily accessible to plants. The distribution of Zn fractions is strongly influenced by soil properties such as pH, organic matter content, clay content, and the abundance of Fe and Al oxides, as well as by land use and management practices (Naik and Das, 2007; Ramzan et al., 2014; Thomas et al., 2018). In highly acidic, weathered soils typical of tea plantations, interactions among these factors play a key role in controlling the transformation of Zn between stable and labile pools.

Tea is a perennial cash crop characterized by intensive management practices and a long production cycle. Numerous long-term studies have shown that prolonged tea cultivation induces cumulative changes in soil chemical properties, particularly progressive soil acidification, depletion of exchangeable base cations, reductions in cation exchange capacity, and alterations in nutrient and trace metal dynamics (Wang et al., 2010; Li et al., 2016; Yan et al., 2018). Evidence from major tea-producing regions in northern Vietnam and East Asia indicates that soil degradation intensifies with plantation age, with more pronounced chemical deterioration observed in plantations older than two decades compared with younger stands (Dang, 2005; Dang, 2007; Wang et al., 2010). Based on the stabilization of soil properties and tea yield reported in previous studies, tea plantations are commonly categorized into short-term systems (<15 years), representing an early transitional stage of soil adjustment, and long-term systems (>25 years), in which accumulated soil chemical changes are well established (Dang, 2005; Yan et al., 2018). Such prolonged cultivation is therefore expected to exert a strong influence on the fractionation and bioavailability of micronutrients, including Zn.

Previous studies have highlighted the importance of organic amendments and integrated nutrient management in maintaining Zn bioavailability by enhancing labile Zn fractions and reducing fixation within Fe and Al oxide complexes (Alloway, 2008; Regmi et al., 2010; Dhaliwal and Dhaliwal, 2019; Singh et al., 2019). However, relatively little is known about how short-term versus long-term tea cultivation, in comparison with adjacent forest soils, influences Zn fractionation and availability under the strongly acidic soil conditions of northern Vietnam. Moreover, the relationships between Zn fractions and key soil properties, including pH, organic carbon, clay content, and Fe and Al oxides, remain poorly documented in this region.

Therefore, the objectives of this study were to (i) characterize the distribution and vertical patterns of Zn fractions in soils under contrasting land uses (long-term tea, short-term tea, and forest), (ii) assess the role of soil properties in regulating Zn partitioning across different soil types, and (iii) determine the relationships between potentially available Zn and specific Zn fractions. The results are expected to provide a scientific basis for soil and nutrient management strategies that can sustain Zn bioavailability and support the long-term productivity and quality of tea cultivation in Vietnam.

2. Materials and methods

2.1. Study area and site

The study was conducted in Tan Cuong commune, Thai Nguyen province, northern Vietnam, a region widely recognized for the production of high-quality green tea, and in Song Cau commune, which was included for comparative purposes. Both communes have a long history of tea cultivation; however, they differ markedly in cultivation practices, productivity, and tea quality. Tan Cuong is regarded as a superior tea production area, characterized by intensive management and consistently high yields and quality, whereas Song Cau represents a more marginal area, where tea productivity and quality are generally lower due to differences in soil conditions and management intensity.

The spatial distribution of the study areas, together with the locations of sampling sites and transect lines in Tan Cuong commune, is shown in Fig. 1. Tan Cuong commune is situated within the floodplain of the Cong River and its adjacent terraces. The landscape is characterized by back marsh lowlands interspersed with natural and former dikes near the river, transitioning to gently undulating river terraces further inland. Topography ranges from flat lowlands to gently sloping terraces with gradients of approximately 5–15°. Tea plantations are distributed across both floodplain margins and sloping terrace lands, while paddy fields predominantly occupy the lower-lying areas farther from the river. Geologically, the area is underlain by Mesozoic schists and sedimentary rocks, including sandstones and mudstones, with recent alluvial deposits occurring along the riverbanks.

In contrast, the Song Cau commune was selected because it represents a different and more weathered soil type (Ultisols) and is characterized by less intensive tea management. Including this site allows for comparison of Zn fractionation and availability under contrasting soil conditions and management practices.

Four tea gardens and two adjacent forest patches were selected in Tan Cuong commune along transect B, approximately perpendicular to the Cong River, as illustrated in Fig. 1. These included TB1 and FB1 located near the river terrace, TB2, TB3, and FB3 situated on undulating former terraces, and TB4 located further inland. An additional tea garden (TS1) was selected in Song Cau commune. Key site characteristics, including topographic position, soil type, tea garden age, and fertilizer application rates, are summarized in Table 1. Information on tea garden age and fertilizer use was obtained through interviews with local farmers.

Based on plantation age and management history, tea plantations were classified into short-term and long-term cultivation categories. Short-term tea cultivation was assigned to the TB1 and TB4 sites, whereas long-term tea cultivation was assigned to the TB2, TB3, and TS1 sites.

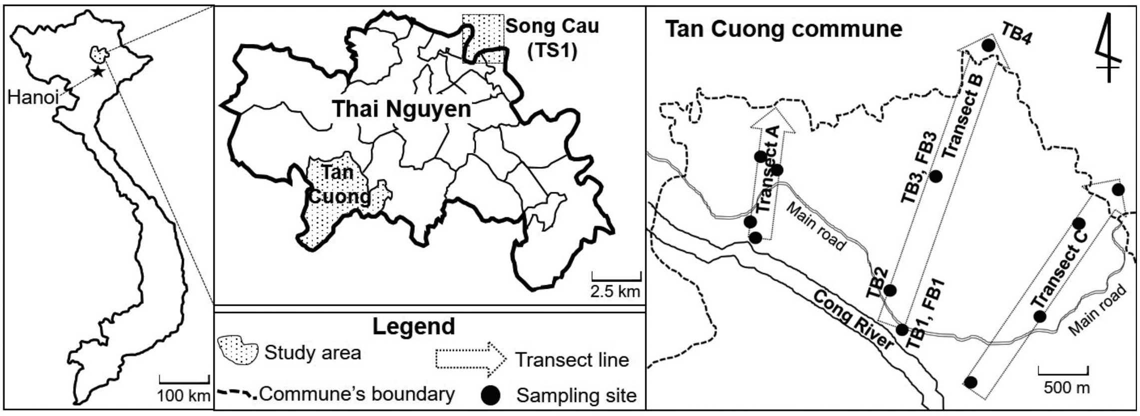

Fig 1. Study area in Tan Cuong and Song Cau communes and transect lines in Tan Cuong commune. The present study focused on transect B as defined by Huu Chien et al. (2019)

Table 1

Information about the study sites

Site	Distance from the river (m)	Topography	Landform	Slope gradient (°)	Soil type	Garden age (yrs)	Annual fertilizer amount			
							N	P_2O_5	K_2O	Organic
							kg ha^{-1}			ton ha^{-1}
TB1	109	flat land	flat	0	Typic Ustipsaments	14	750	220	150	26
TB2	212	previous dike	undulating	12	Typic Dystrustepts	44	356	433	63	11
TB3	1,340	previous dike	undulating	13	Typic Dystrustepts	35	859	427	240	29
TB4	2,380	previous dike	undulating	6	Typic Dystrustepts	6	286	210	114	24
FB1	109	flat land	flat	0	Typic Ustipsaments	–	–	–	–	–
FB3	1,340	previous dike	undulating	3	Typic Dystrustepts	–	–	–	–	–
TS1	–	rolling hill	hill slope	24	Typic Paleustults	30	575	200	417	0

According to Huyen (2013), the recommended fertilizer application rate for tea cultivation in Vietnam is 300 kg N, 150 kg P_2O_5, and 150 kg K_2O per hectare. In the Tan Cuong area, chemical fertilizers-primarily urea and various NPK compound formulations-are commonly applied by surface broadcasting seven to nine times per year, mainly during the rainy season. Homemade organic manure, when available, is applied between tea hedges, and plant residues generated during pruning and harvesting are typically retained in the field. The application rates of nitrogen and phosphorus in both communes generally exceeded the recommended levels, whereas potassium inputs were comparatively lower at the TB1, TB2, and TB4 sites.

Based on results from a previous study conducted in the same area (Huu Chien et al., 2020), soils at the TB1 and FB1 sites were classified as Typic Ustipsamments of Entisols according to the USDA Soil Taxonomy (Soil Survey Staff, 2014). Soils at the TB2, TB3, FB3, and TB4 sites were classified as Typic Dystrustepts of Inceptisols. In contrast, the soil at the TS1 site in Song Cau commune was classified as Typic Paleustults of Ultisols.

2.2. Soil sampling and analysis

At each study site, a representative soil pit was prepared and described. Soil samples were collected by horizon and air-dried for analysis. Soil pH was measured in both water and 1 M KCl at a 1:5 soil-to-solution ratio using a glass electrode pH meter (Horiba F-21). Total carbon (TC) was determined using a CN analyzer (JM 1000, J-Science Lab). Cation exchange capacity (CEC) was calculated from the NH_4^+ retained after extraction with 1 M ammonium acetate at pH 7.0 and displaced with 10% NaCl by steam distillation. Clay content was determined by the pipette method.

Free oxides of Fe, Al, and Si (Fe_d, Al_d, Si_d) were extracted using the dithionite-citrate (DC) method, while poorly crystalline (amorphous) forms (Fe_o, Al_o, Si_o) were extracted using acid ammonium oxalate [$(NH_4)_2C_2O_4$]. The Fe, Al, and Si contents were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES; ICPS 7510, Shimadzu). Ratios of Fe_o/Fe_d, Al_o/Al_d, and Si_o/Si_d were calculated to indicate degrees of weathering.

The distribution of Zn among chemical fractions was determined by sequential extraction following Iwasaki and Sakurai (1996). For each 3.0 g soil sample, the following steps were conducted sequentially:

- Step 1: Exchangeable Zn (Ex-Zn): extracted with 1 M CH_3COONH_3 (pH 7.0) for 2 hours at a 1:10 soil-to-solution ratio.
- Step 2: Acid-soluble Zn (Aci-Zn): extracted with 25 g L^{-1} CH_3COOH (pH 2.6) for 6 hours (1:10 ratio).
- Step 3: Mn oxide-bound Zn (MnO-Zn): extracted with 0.1 M $NH_2OH{\cdot}HCl$ + HNO_3 (pH 2.0) for 0.5 hour (1:10 ratio).
- Step 4: Organic matter-bound Zn (OM-Zn): extracted with 0.1 M $Na_4P_2O_7$ (pH 10.0) for 24 hours at 1:50 ratio.
- Step 5: Fe oxide-bound Zn (FeO-Zn): extracted with 0.175 M $(NH_4)_2C_2O_4$ + 0.1 M $H_2C_2O_2$ + 0.1 M ascorbic acid (pH 3.1) at 1:50 ratio, shaken for 4 hours and subsequently heated in a boiling water bath for 0.5 hour with occasional stirring.
- Step 6: Residual Zn (Res-Zn): determined by digestion in H_2SO_2-HNO_3-$HClO_4$ (1:5:20).

The Zn concentrations in extracts were quantified using atomic absorption spectrometry (Shimadzu AA-610S). Total Zn was obtained from complete digestion in the same mixed acid. Zn recovery was calculated as the ratio of the sum of fractions to the total digestion value.

Potentially available Zn was assessed by extraction with a DTPA-TEA-$CaCl_2$ solution (0.005 M DTPA, 0.01 M $CaCl_2$, 0.1 M TEA; pH 7.3). Ten grams of air-dried soil was shaken with 20 mL of the extractant (soil-to-solution ratio 1:2, w/v) for 2 hours at room temperature. The leachate was then filtered, and Zn contents were determined by ICP-AES (Shimadzu ICPS-7510).

2.3. Statistical analysis

Pearson correlation analysis was conducted to examine relationships between Zn fractions and selected soil properties, including soil pH, total carbon content, clay content, and different forms of Fe and Al oxides.

Differences in Zn fractions between tea cultivation durations (short-term vs. long-term) and between land uses (tea vs. forest) were evaluated using independent-sample t-tests. These comparisons were performed using all available samples for each group (short-term tea, n = 7; long-term tea, n = 15; tea soils, n = 22; forest soils, n = 10). To assess the robustness of the land-use comparison, an additional independent-sample t-test was conducted after excluding samples from the TS1 site (Ultisols), which represents a more weathered soil developed under distinct management conditions; in this restricted dataset, tea soils (n = 17) were compared with forest soils (n = 10).

Differences in Zn fractions among soil types (Entisols, Inceptisols, and Ultisols) were assessed using one-way analysis of variance (ANOVA). When significant effects were detected, mean separation was performed using LSD test.

All statistical analyses were conducted using IBM SPSS Statistics 26.0 software, and statistical significance was defined at $p < 0.05$.

3. Results

3.1. Physicochemical properties and the contents of oxide-hydroxide of soil

The physicochemical properties of the studied soils are presented in Table 2. Soil pH(H_2O) values across all sites ranged from 3.4 to 5.9 and generally increased with depth. Tea-growing soils (TB and TS sites) were characterized by strongly acidic conditions throughout the profiles, whereas forest soils (FB sites) showed relatively higher pH values, particularly in subsurface horizons.

Total carbon (T-C) and total nitrogen (T-N) contents were highest in surface horizons and decreased with depth at all sites. Long-term tea plantations (TB2 and TB3) exhibited markedly higher T-C and T-N contents in surface horizons compared with short-term tea sites and forest soils. Cation exchange capacity (CEC) varied widely among sites and horizons, with higher values generally observed in horizons with greater organic carbon contents.

Exchangeable Ca, Mg, K, and Na contents were low across all profiles, while exchangeable Al dominated the exchange complex, particularly in tea soils. Clay contents ranged from 8 to 50%, with sandy textures prevailing in TB1 and FB1, whereas finer textures were observed in TB2, TB3, and TS1 soils. The contents of amorphous (Si_o, Al_o, Fe_o) and crystalline (Si_d, Al_d, Fe_d) oxide-hydroxides varied among sites and generally increased with depth.

3.2. Zn fractions, total Zn, and potentially available Zn in soils

Average Zn recovery after sequential extraction was 105.5%, indicating acceptable analytical performance. Zinc fractionation results are presented as six operationally defined fractions together with total Zn and DTPA-extractable Zn (Table 3).

Across all soils and depths, Zn was predominantly associated with the residual fraction, followed by Fe oxide-bound (FeO-Zn), organic matter-bound (OM-Zn), acid-soluble (Aci-Zn), Mn oxide-bound (MnO-Zn), and exchangeable (Ex-Zn) fractions. This general fractionation pattern was consistent among land uses and soil types.

Vertical distributions of Zn fractions varied systematically with soil depth and soil type. Labile fractions, including Ex-, Aci-, and MnO-Zn, were consistently enriched in surface horizons and declined with depth across all profiles. In Inceptisols and Ultisols, OM-Zn was also concentrated in surface soils and decreased downward, whereas in Entisols, OM-Zn showed relatively lower surface enrichment and a more even or increasing distribution with depth. FeO-Zn tended to increase with depth in Entisols but declined in Inceptisols and Ultisols, while the residual fraction generally increased with depth in Entisols and Ultisols and remained relatively stable in Inceptisols.

Within the Tan Cuong commune, soils under long-term tea cultivation (TB2 and TB3) generally exhibited higher concentrations of most Zn fractions, total Zn, and DTPA-Zn in surface horizons compared with soils under short-term tea cultivation (TB1 and TB4). These contrasts were most pronounced for the more labile Zn fractions and diminished with increasing depth.

Table 2
Physicochemical properties and Si, Al, and Fe oxide-hydroxide contents of soils

Site	Depth (cm)	Horizon	pH (H_2O)	T-C	T-N	CEC	Exchangeable cations					Clay	Silt	Sand	Oxide-hydroxides					
				g kg^{-1}			Ca	Mg	K	Na	Al	%			Si_o	Al_o	Fe_o	Si_d	Al_d	Fe_d
							cmol$_c$ kg^{-1}								g kg^{-1}					
TB1	0–3	A	3.7	33.5	1.98	9.07	2.11	0.59	0.31	0.05	1.17	13	10	77	0.08	0.93	2.78	0.34	1.30	6.16
	3–8	BA	3.6	13.0	0.67	5.73	0.27	0.08	0.07	0.03	1.50	11	10	79	0.04	0.84	2.02	0.33	1.26	6.14
	8–20	Bw	3.8	8.2	0.44	4.52	0.13	0.04	0.07	0.03	1.17	11	11	78	0.04	0.80	1.68	0.31	1.25	5.84
	20–57	C1	3.9	4.6	0.27	3.51	0.09	0.02	0.03	0.02	1.17	13	12	75	0.04	0.62	1.51	0.39	1.30	6.56
	57–80^{+}	C2	3.9	3.7	0.23	4.22	0.12	0.03	0.04	0.03	1.42	17	15	68	0.04	0.67	1.25	0.38	1.73	9.77
TB2	0–3	A	4.1	16.2	1.33	14.3	1.17	0.25	0.17	0.04	5.17	32	21	47	0.07	1.70	3.31	0.40	2.67	14.7
	3–11	AB	4.3	12.7	1.02	28.1	1.01	0.10	0.07	0.10	5.50	33	22	45	0.05	1.75	3.10	0.41	2.88	17.3
	11–25	2Bw1	4.3	9.5	0.92	39.0	0.88	0.08	0.08	0.03	9.08	50	21	28	0.07	2.45	2.17	0.51	3.78	22.0
	25–54	3Bw2	4.3	13.0	0.87	24.4	0.29	0.03	0.04	0.03	4.75	28	20	52	0.06	1.81	4.70	0.34	2.77	15.5
	54–70^{+}	4Bw3	4.2	9.0	0.71	14.2	0.30	0.04	0.05	0.03	6.42	34	22	44	0.05	1.89	3.20	0.35	3.56	21.1
TB3	0–2	A	3.4	99.9	7.73	40.6	1.58	0.35	0.46	0.06	5.42	33	27	40	0.04	3.25	5.97	0.70	3.98	14.5
	2–12	BA	3.4	19.1	1.32	18.7	0.56	0.13	0.25	0.04	10.1	29	21	50	0.03	3.35	3.76	0.34	3.76	17.0
	12–30	Bw1	3.6	10.8	0.80	13.8	0.26	0.06	0.10	0.03	6.83	29	21	50	0.02	2.05	2.61	0.41	3.13	15.4
	30–52	Bw2	3.6	13.5	0.97	14.6	0.23	0.06	0.09	0.03	6.58	22	15	63	0.03	2.33	3.02	0.27	3.29	15.7
	52–65^{+}	Bw3	3.6	14.1	0.99	12.0	0.24	0.05	0.05	0.03	5.50	28	23	49	0.02	1.86	3.65	0.22	2.40	11.9
TB4	0–2	A	4.4	10.4	0.80	9.89	2.22	0.27	0.18	0.06	2.33	34	23	43	0.01	0.70	0.49	0.47	1.86	19.1
	2–40^{+}	C	4.4	5.3	0.38	7.46	0.41	0.04	0.06	0.03	3.75	33	23	44	0.01	0.67	0.40	0.51	1.89	18.6
TS1	0–2	A1	4.1	26.4	1.82	16.1	0.45	0.14	0.17	0.02	6.58	38	19	43	0.02	2.27	2.72	0.34	4.16	22.9
	2–9	A2	4.0	20.2	1.35	15.5	0.18	0.07	0.09	0.02	6.75	39	19	42	0.00	2.21	2.77	0.22	3.82	21.1
	9–22	Bw1	4.2	9.3	0.60	13.2	0.30	0.03	0.04	0.02	5.67	45	19	37	0.03	1.61	2.06	0.29	4.13	24.6
	22–37	Bt	4.3	8.6	0.56	13.9	0.14	0.04	0.05	0.01	4.92	47	17	36	0.03	1.48	1.64	0.28	4.42	26.7
	37–50^{+}	Bw2	4.6	10.4	0.67	14.7	0.22	0.03	0.04	0.02	4.58	49	19	32	0.05	1.38	1.30	0.47	4.47	25.1
FB1	0–4	A1	4.5	20.9	1.02	6.38	0.75	0.13	0.14	0.01	1.01	13	15	72	0.04	0.96	2.31	0.25	1.48	7.90
	4–15	A2	5.8	15.1	0.75	5.26	0.61	0.10	0.09	0.01	1.01	14	16	70	0.05	0.98	2.52	0.22	1.55	8.58
	15–24	AB	5.7	10.7	0.56	4.46	0.37	0.06	0.12	0.01	1.01	15	10	75	0.05	0.89	2.50	0.24	1.49	8.54
	24–52	Bw	5.9	6.3	0.26	2.93	0.36	0.02	0.10	0.01	0.67	14	11	74	0.06	0.73	2.28	0.22	1.23	7.38
	52–85^{+}	C	5.4	1.7	0.07	1.61	0.11	0.00	0.10	0.00	0.42	8	7	85	0.02	0.37	1.12	0.14	0.70	4.16
FB3	0–4	A	4.3	61.2	2.90	14.8	2.80	0.59	0.24	0.06	3.09	25	27	49	0.02	1.34	2.87	0.35	2.64	14.4
	4–18	AB	4.5	16.7	0.84	7.12	0.29	0.06	0.12	0.02	2.80	23	25	51	0.06	1.13	3.63	0.37	2.32	16.6
	18–30	Bw1	4.8	11.6	0.58	7.53	0.55	0.11	0.10	0.02	2.46	25	25	50	0.05	0.94	3.68	0.26	2.24	14.7
	30–42	Bw2	4.8	11.7	0.62	8.37	0.65	0.08	0.10	0.02	2.89	29	20	51	0.05	0.95	3.63	0.42	2.45	14.4
	42–50^{+}	Bw3	4.7	11.8	0.56	8.15	0.55	0.05	0.10	0.02	2.80	26	23	51	0.06	1.06	3.70	0.31	2.39	13.8

Table 3
The contents of Zn fractions, total Zn, and DTPA Zn in soils

Site	Sample	Depth (cm)	Ex-	Aci-	MnO-	OM-	FeO-	Res-	Sum of fractions	Total	Recovery (Sum/total) %	DTPA mg kg⁻¹
			mg kg⁻¹									
TB1	TB1-A	0–3	0.40	1.05	0.82	2.72	6.87	19.60	31.47	28.60	110.0	1.47
	TB1-BA	3–8	0.46	0.58	0.40	2.49	5.14	20.68	29.74	29.33	101.4	0.72
	TB1-Bw	8–20	0.31	0.66	0.36	2.69	6.35	21.98	32.33	32.57	99.3	0.71
	TB1-C1	20–57	0.26	0.65	0.41	2.62	6.52	26.90	37.36	36.70	101.8	0.61
	TB1-C2	57–80⁺	0.25	0.89	0.34	2.55	7.24	34.69	45.95	44.16	104.1	0.75
TB2	TB2-A	0–3	2.06	2.15	1.20	3.99	5.12	40.56	55.08	49.02	112.4	3.63
	TB2-AB	3–11	0.28	1.37	0.67	4.63	4.26	40.86	52.08	49.87	104.4	2.11
	TB2-2Bw1	11–25	0.17	0.85	0.38	3.07	4.05	50.02	58.55	50.71	115.5	0.56
	TB2-3Bw2	25–54	0.13	0.47	0.32	3.27	3.52	35.99	43.70	37.61	116.2	0.38
	TB2-4Bw3	54–70⁺	0.13	0.45	0.21	2.66	3.13	39.40	45.98	43.08	106.7	0.32
TB3	TB3-A	0–2	0.42	1.14	0.97	4.67	9.10	16.18	32.47	33.21	97.8	2.57
	TB3-BA	2–12	0.23	0.94	0.58	2.42	3.54	21.48	29.19	24.51	119.1	1.11
	TB3-Bw1	12–30	0.07	0.39	0.15	1.89	3.00	25.41	30.92	28.26	109.4	0.68
	TB3-Bw2	30–52	0.10	0.41	0.18	2.45	5.18	23.91	32.22	29.06	110.9	0.69
	TB3-Bw3	52–65⁺	0.14	0.40	0.17	1.71	3.04	16.73	22.18	21.07	105.3	0.69
TB4	TB4-A	0–2	0.50	2.79	1.24	1.93	4.10	8.60	19.16	18.66	102.6	4.82
	TB4-C	2–40⁺	0.02	0.42	0.17	0.36	2.47	8.61	12.06	12.01	100.4	0.53
TS1	TS1-A1	0–2	0.07	0.62	0.40	3.45	5.11	17.10	26.76	23.83	112.3	1.31
	TS1-A2	2–9	0.04	0.58	0.25	5.42	4.29	17.73	28.31	22.08	128.2	0.76
	TS1-Bw1	9–22	0.00	0.24	0.05	1.59	2.05	19.73	23.66	23.15	102.2	0.25
	TS1-Bt	22–37	0.00	0.18	0.04	1.90	2.19	20.26	24.57	24.81	99.0	0.17
	TS1-Bw2	37–50⁺	0.00	0.11	0.01	1.43	4.34	24.56	30.45	25.68	118.6	0.07
FB1	FB1-A1	0–4	0.44	1.57	0.79	3.66	13.72	29.22	49.40	47.76	103.4	3.14
	FB1-A2	4–15	0.33	1.51	0.67	3.13	14.00	28.74	48.38	51.19	94.5	2.79
	FB1-AB	15–24	0.18	1.02	0.35	3.17	10.30	29.25	44.27	46.28	95.7	1.55
	FB1-Bw	24–52	0.04	0.29	0.12	2.34	12.39	24.55	39.73	39.06	101.7	0.29
	FB1-C	52–85⁺	0.03	0.19	0.04	1.34	7.97	15.12	24.68	25.68	96.1	0.11
FB3	FB3-A	0–4	0.80	2.19	1.50	4.22	5.60	22.32	36.62	37.02	98.9	7.65
	FB3-AB	4–18	0.07	0.67	0.23	2.09	3.64	21.60	28.30	27.01	104.8	1.49
	FB3-Bw1	18–30	0.12	0.77	0.20	2.01	3.07	25.00	31.17	25.98	120.0	1.69
	FB3-Bw2	30–42	0.04	0.45	0.09	1.64	2.61	26.24	31.08	30.70	101.2	0.99
	FB3-Bw3	42–50⁺	0.07	0.37	0.23	0.99	2.65	26.62	30.94	27.92	110.8	0.62

In contrast, Ultisols at the TS1 site in Song Cau commune displayed consistently low contents of labile Zn fractions and DTPA-Zn throughout the profiles, particularly in subsoil horizons, whereas residual Zn constituted a substantial proportion of total Zn. This pattern reflects strong Zn stabilization in more weathered soils.

Forested soils (FB1 and FB3) generally showed higher Zn contents in surface horizons than their adjacent tea soils, particularly for FeO-Zn and DTPA-Zn, while total Zn contents were broadly comparable. These differences were most evident in topsoil layers and decreased with depth.

3.3. Correlations between Zn fractions and potentially available Zn contents and selected soil properties

Correlation analysis (Table 4) revealed significant associations between Zn fractions, total Zn, DTPA-Zn, and soil properties. FeO-Zn was positively correlated with soil pH (r = 0.47**), but negatively correlated with clay content (r = –0.58**) and crystalline Fe oxides (Fe_d, r = –0.62**). OM-Zn was positively correlated with cation exchange capacity (CEC, r = 0.46**), total carbon (TC, r = 0.51**), amorphous Fe oxides (Fe_o, r = 0.41*), and amorphous Al oxides (Al_o, r = 0.42*). Res-Zn showed strong positive correlation with amorphous Si oxides (Si_o, r = 0.65**) and the Si_o/Si_d ratio (r = 0.42*).

DTPA-Zn content was strongly and positively correlated with Ex- (r = 0.61**), Aci- (r = 0.90**), and MnO- (r = 0.90**) fraction contents. It also correlated positively, though less strongly, with OM-Zn (r = 0.44*) and TC (r = 0.36*) contents. Total Zn showed significant positive correlation with Si_o (r = 0.58**) and total Mn (r = 0.61**).

4. Discussion

Slightly elevated recovery values (> 115% in a few samples) may reflect analytical uncertainties inherent to sequential extraction procedures, including incomplete removal of extractants between successive steps, partial redistribution of Zn among operationally defined fractions, and minor instrumental or spectral interferences during ICP-AES measurement (Tessier et al., 1979; Sutherland, 2010). Similar deviations have been widely reported in studies employing multi-step extraction schemes and are generally considered acceptable when mean recoveries remain close to 100% (Bacon and Davidson, 2008), as observed in the present study.

Across all soils, Zn was predominantly associated with the residual fraction, followed by FeO-, OM-, Aci-, MnO-, and Ex- fractions. This fractionation order agrees well with previous studies on Zn (Okoli et al., 2016; Indira et al., 2019; Sanjib et al., 2022) and with findings for Mn in the same region (Huu Chien et al., 2024), indicating that most Zn is retained in geogenic and relatively immobile pools, while only a smaller proportion resides in potentially bioavailable forms.

Table 4
Pearson correlation between Zn fraction and DTPA Zn contents and selected soil properties (n=32)

	Ex-	Aci-	MnO-	OM-	FeO-	Res-	Total	DTPA-
pH	–0.16	0.00	–0.17	–0.16	0.47**	0.10	0.28	0.03
CEC	0.07	0.10	0.23	0.46**	–0.25	0.34	0.18	0.08
TC	0.22	0.25	0.49**	0.51**	0.14	–0.21	–0.02	0.36*
Clay	–0.11	–0.10	–0.11	0.03	–0.58**	0.11	–0.21	–0.09
Fe_d	–0.19	–0.12	–0.16	–0.03	–0.62**	0.03	–0.29	–0.08
Fe_o	0.12	–0.01	0.15	0.41*	–0.06	0.22	0.17	0.09
Fe_o/Fe_d	0.19	0.04	0.20	0.24	0.48**	0.04	0.27	0.06
Al_d	–0.14	–0.20	–0.13	0.18	–0.50**	0.11	–0.18	–0.12
Al_o	0.02	–0.04	0.10	0.42*	–0.25	0.15	–0.01	–0.03
Al_o/Al_d	0.23	0.12	0.27	0.46**	0.30	0.14	0.26	0.05
Si_o	0.24	0.01	0.04	0.05	0.19	0.65**	0.58**	–0.11
Si_d	0.17	0.23	0.31	0.09	–0.25	0.07	–0.01	0.17
Si_o/Si_d	0.07	–0.06	–0.09	–0.06	0.43*	0.42*	0.50**	–0.12
Total Mn	0.18	0.20	0.20	0.30	0.93**	0.17	0.61**	0.11
DTPA–	0.61**	0.90**	0.90**	0.44*	0.24	0.05	0.23	1.00

* and ** indicate the significance of correlations at 0.05 and 0.01 levels, respectively

Statistical comparisons consistently identified FeO-Zn as the only Zn fraction showing significant differences among land use types, tea cultivation duration, and soil orders. FeO-Zn contents were significantly lower in long-term tea plantations than in short-term plantations (4.13 vs. 5.53 mg kg^{-1}, $p < 0.05$), indicating a cumulative effect of prolonged tea cultivation on the redistribution or depletion of Zn associated with amorphous Fe oxides under strongly acidic conditions. This result highlights FeO-Zn as a particularly sensitive indicator of long-term soil chemical alteration in tea monoculture systems.

Significant differences in FeO-Zn were also observed among soil types. When both tea and forest soils were considered, Entisols contained substantially higher FeO-Zn (9.05 mg kg^{-1}) than Inceptisols (4.00 mg kg^{-1}) and Ultisols (3.60 mg kg^{-1}), while no significant difference was detected between the latter two. A similar decreasing trend from Entisols to Inceptisols and Ultisols was evident when only tea soils were analyzed. This pattern is consistent with progressive pedogenic development, whereby increasing weathering intensity promotes the transformation of amorphous Fe oxides into more crystalline forms with lower Zn sorption capacity.

Land use also influenced FeO-Zn. Tea soils exhibited significantly lower FeO-Zn contents than adjacent forest soils (4.57 vs. 7.60 mg kg^{-1}, $p < 0.05$), and this difference remained statistically significant after excluding the TS1 site. The consistently higher FeO-Zn in forest soils likely reflects more stable Fe oxide pools under less disturbed conditions, whereas intensive tea cultivation enhances soil acidification and accelerates Fe oxide crystallization, thereby reducing the retention of Zn in Fe-oxide-bound forms.

The fact that statistically significant differences were confined to FeO-Zn, while other Zn fractions showed no clear separation among groups, underscores the particular sensitivity of this fraction to long-term soil management and weathering processes. Fe oxides are widely recognized as major sorbents for Zn in acidic soils, and changes in their abundance and crystallinity directly control Zn buffering capacity (Ghanem and Mikkelsen, 1988; Li et al., 2012). Accordingly, FeO-Zn emerges as the most responsive Zn pool to prolonged tea cultivation and pedogenic development in northern Vietnam.

Although no statistically significant differences were detected for Ex-, Aci-, MnO-, OM-, or DTPA-Zn among land uses or cultivation durations, several consistent descriptive patterns provide useful context for understanding Zn dynamics. Ex-, Aci-, and MnO- fractions and DTPA Zn were generally enriched in surface horizons and declined with depth, suggesting that fertilizer inputs and organic matter accumulation play an important role in Zn enrichment of topsoils under tea cultivation.

The enrichment of OM-Zn in surface horizons of Inceptisols and Ultisols further reflects the contribution of organic matter inputs to Zn retention, whereas the contrasting pattern observed in Entisols may be related to their lower clay content and surface instability under frequent flooding. These trends, while not statistically significant, suggest that organic matter dynamics and soil texture modulate the partitioning of Zn into relatively labile pools.

Surface soils under forest tended to show higher Zn contents across most fractions and higher DTPA-Zn than adjacent tea soils, and long-term tea plantations generally exhibited higher surface Zn contents than short-term plantations. These tendencies, interpreted cautiously, indicate that continuous organic inputs from forest litter or prolonged management practices in tea systems may favor the accumulation of Zn in surface soils, particularly in exchangeable and organically bound forms.

Depth-related variations in Zn fractions were closely linked to pedogenic indicators. In Inceptisols and Ultisols, the decline of FeO-Zn with depth corresponded to reductions in amorphous Fe, Al, and Si oxides and to lower Fe_o/Fe_d, Al_o/Al_d, and Si_o/Si_d ratios, reflecting progressive weathering and oxide crystallization. Conversely, the increase of Res-Zn with depth in Ultisols coincided with higher clay content, CEC, and weathering indices, suggesting stronger fixation of Zn in stable mineral forms in subsoil horizons.

In the TB2 profile, abrupt vertical changes in FeO-Zn, Al_o, Fe_o, Al_d, and Fe_d contents between successive Bw horizons coincided with marked morphological contrasts, indicating stratified pedogenic development. These features likely reflect episodic sediment inputs or management-induced disturbance that locally modified Fe oxide transformation pathways and Zn retention capacity.

Site-specific contrasts further emphasize the combined influence of land use and pedogenesis. TB2 soils exhibited exceptionally high Ex-, Aci-, MnO-, and OM-Zn but relatively low FeO-Zn, consistent with intensive organic amendments under long-term tea cultivation combined with lower weathering intensity. In contrast, TB4 soils showed the lowest FeO-Zn contents, likely reflecting excavation activities that accelerated the transformation of amorphous to crystalline Fe oxides. Ultisols at TS1 were characterized by extremely low Ex-, Aci-, MnO-, and DTPA-Zn contents together with modest OM-, FeO-, and Res-Zn, pointing to Zn-poor parent material combined with advanced weathering.

Correlation analysis provided further insight into the mechanisms governing Zn distribution. The positive correlation between FeO-Zn and soil pH ($r = 0.47^{**}$) indicates greater stability of FeO-Zn under less acidic conditions. The significant association between OM-Zn and CEC ($r = 0.46^{**}$) supports the role of organic matter in enhancing charge retention and cation exchange capacity (Huu Chien et al., 2019). In addition, positive correlations between T-C and MnO-, OM-, and DTPA-Zn contents suggest that organic inputs promote the mobilization of Zn from stable pools through organic acid complexation.

Negative correlations between clay content and FeO-Zn ($r = -0.58^{**}$) and between Fe_d and FeO-Zn ($r = -0.62^{**}$) emphasize the contrasting sorption behavior of amorphous versus crystalline Fe oxides. Amorphous oxides possess higher surface area and reactivity (Music and Ristic, 1992; Goldberg et al., 2001), but their transformation during soil aging reduces Zn retention. The positive relationships between Al_o content and Al_o/Al_d ratio and OM-Zn content further highlight the role of organic matter in slowing weathering processes and maintaining Zn in less stable, more reactive pools.

Comparison with Mn fractionation results from the same tea soils (Huu Chien et al., 2024) reveals contrasting biogeochemical controls. Zn fractions were primarily governed by organic carbon and CEC, reflecting the behavior of Zn^{2+} as a non-redox-active cation whose mobility is strongly influenced by proton competition and organic complexation. In contrast, Mn fractions were closely linked to Fe- and Al-oxides and redox-sensitive pools, consistent with the redox-active nature of Mn.

Although both elements were predominantly retained in the residual fraction, Zn showed a relatively greater proportion in exchangeable and organically bound forms, indicating higher sensitivity to acidification and organic inputs. Mn, by contrast, was more strongly associated with oxide-bound pools, highlighting its regulation by Fe and Al oxides and potential mobilization under reducing conditions. These differences suggest that soil acidification in tea plantations enhances Zn availability mainly by weakening surface adsorption and promoting organic chelation, whereas Mn availability is more strongly controlled by oxide dissolution and redox processes.

Finally, the strong correlations between DTPA-Zn and Ex- ($r = 0.61^{**}$), Aci- ($r = 0.90^{**}$), MnO- ($r = 0.90^{**}$), and OM-Zn ($r = 0.44^{*}$) confirm that these fractions represent the main contributors to Zn bioavailability. The positive association with T-C ($r = 0.36^{*}$) further underscores the central role of organic matter in sustaining Zn solubility in these acidic tea soils.

5. Conclusions

This study demonstrates that Zn in tea garden soils of northern Vietnam is predominantly associated with the Res-fraction, followed by FeO-, OM-, Aci-, MnO-, and Ex- fractions, reflecting advanced weathering under strongly acidic conditions. Although Res-Zn accounted for the largest proportion of total Zn, DTPA-Zn was closely related to the Ex-, Aci-, MnO-, and OM- fractions, indicating that Zn availability is primarily governed by labile pools rather than total Zn content.

Land use and soil type exerted clear influences on Zn fractionation patterns. Descriptive comparisons indicated that forest soils and long-term tea plantations tended to contain higher contents of labile Zn fractions than short-term tea plantations. However, statistical analyses consistently identified FeO-Zn as the only fraction exhibiting significant differences among tea cultivation duration, soil types, and land uses. Specifically, FeO-Zn contents were significantly lower in long-term tea plantations and in tea soils compared with adjacent forest soils, highlighting the sensitivity of this fraction to prolonged tea monoculture.

Soil organic matter and cation exchange capacity were key factors enhancing labile Zn pools, whereas progressive soil acidification and the transformation of amorphous to more crystalline Fe/Al oxides under long-term cultivation reduced Zn retention in Fe-associated forms. Compared with Mn, which was largely regulated by oxide interactions and redox conditions, Zn dynamics were more strongly controlled by organic complexation and exchange processes.

Conflict of interest

The authors declare no competing financial interests or personal relationships that could have influenced the work reported in this paper. This research did not involve human or animal subjects.

Author Contribution

Huu Chien Hoang – Conceptualization, Methodology, Data curation, Writing – original draft, Writing – review & editing. **Kozo Iwasaki** – Conceptualization, Funding acquisition, Data curation. **Sota Tanaka** – Supervision, Validation, Data curation.

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