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Assessment of Zinc Sources on Soil Properties and Fractional Distribution in Clay Loam Soils of Rice Cultivation Zones of Tamilnadu

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ABSTRACT

The study aimed to assess the efficiency of various zinc sources on soil properties and the bioavailability of zinc in clay loam soil, incubated at ambient temperature for 90 days. The results indicated that zinc fractions, *viz.,* Water Soluble-Exchangeable, Complex, Organic bound, Occluded, Residual, Total and DTPA-extractable zinc, significantly increased up to 45 days, followed by a decline as the incubation period progressed across all treatments. The release pattern of zinc from different sources revealed that zinc concentration was highest on the first day across all treatments, and then gradually decreased with time. Among the zinc sources, the application of zinc at 5 kg ha^{-1} through $ZnSO_4$ resulted in significantly higher levels of available zinc and its fractions, followed by zinc lysinate. Additionally, the application of $ZnSO_4$ at 5 kg ha^{-1} led to the greatest reduction in soil pH (7.02), while also recording the highest electrical conductivity (0.34 dS m^{-1}).

Key words: *Zn Sources, Fractions, Release pattern, Soil properties, Rice.*

Introduction

Zinc (Zn) is vital for plant health, serving as a structural component and regulatory co-factor in key biochemical processes such as carbohydrate metabolism, photosynthesis, starch synthesis, protein and auxin metabolism, pollen formation, membrane integrity, and disease resistance. It is the second most limiting nutrient after nitrogen. In rice, Zn deficiency leads to stunted growth, delayed maturity, poor root development, shriveled grains, and yield losses of 10–60% (Kandil *et al.*, 2022).

In Tamil Nadu, a major rice-growing region, Zn availability strongly affects yield and quality. Soil factors such as pH, organic matter, redox conditions, and nutrient interactions influence Zn dynamics. In clay loam soils, high clay adsorption restricts Zn availability, making fertilization essential. Zn occurs in soils in fractions such as water-soluble, exchangeable, organic-bound, occluded, and residual, each varying in plant availability. Over time, Zn bioavailability declines, and many synthetic Zn forms (ZnO, ZnS, $ZnCO_3$, $Zn_3(PO_4)_2.4H_2O$) are poorly available (Okoli *et al.*, 2016).

Fertilizer efficiency depends on Zn source, soil properties, and environment. Common fertilizers include zinc sulfate ($ZnSO_4$) and chelates (e.g., zinc lysinate). This study evaluates different Zn sources in clay loam soils of Tamil Nadu by incubating soils for 90 days. Changes in pH, EC, and Zn fractions (water-soluble-exchangeable, complexed, organic, occluded, residual, total, and DTPA-extractable) will be assessed. Results will guide Zn management strategies to improve Zn bioavailability and rice productivity.

Materials and Methods

An incubation study was conducted in 2023 at the Ph.D. Laboratory, Faculty of Agriculture, Annamalai University. Surface soil samples (0–15 cm) were collected from a farmer's field in Thiruvalaputhur village, Mayiladuthurai district, air-dried, ground, and sieved (<2 mm). Initial analysis showed slightly alkaline pH (7.78), non-saline EC (0.31 dS m^{-1}), OC (5.92 g kg^{-1}), low N (166 kg ha^{-1}), medium P (13 kg ha^{-1}) and K (176.4 kg ha^{-1}), and Zn deficiency (0.52 mg kg^{-1}).

The experiment followed a completely randomized design with five treatments: T_1 – control, T_2 – zinc sulfate, T_3 – zinc HEDP, T_4 – zinc oxide, and T_5 – zinc lysinate, replicated thrice. A total of 75 cups, each containing 200 g soil, were used. Zinc sources were applied at 5 kg Zn ha^{-1}, mixed thoroughly, and moisture maintained at field capacity with double-distilled water. Incubation lasted 90 days with soil sampling at 1, 30, 45, 60, and 90 days.

Samples were air-dried, sieved, and analyzed for pH (Potentiometry, Jackson, 1973), EC (Conductometry, Jackson, 1973), DTPA-extractable Zn (AAS, Lindsay and Norwell, 1978), and Zn fractions using the sequential method of Sarkar & Deb (1982) given in Table 1. Data were statistically analyzed as per Panse and Sukhatme (1985).

Result and Discussion

Water Soluble plus exchangeable zinc fractions (WS-Ex Zn)

The result showed among the different sources of zinc, the applications of $ZnSO_4$ (T_2) recorded higher WS-Ex Zn at throughout the incubation which recorded the values of 0 (0.96), 30 (1.90) and 45 (3.95) day of incubation which was followed by Lycinate (0.92, 1.60 and 3.59 mg kg^{-1}) respectively. It evidenced from the Fig. 1, that raising WS-Ex Zn due to different zinc sources, the trend in decreasing order was $ZnSO_4$> zinc lysinate > Zn HEDP > Zn oxide > control. The peak for WS-Ex zinc was observed on the 45th day across all treatments, including the control (Fig. 1). Among all the zinc fractions studied, the concentration and per cent contribution of WSEx-Zn fraction to total zinc was the lowest. The highest percent contribution to total zinc (%) recorded in the application of $ZnSO_4$ (T_2) and is in agreement with the findings of Bairagi *et al.* (2020) and Mondal *et al.* (2024).

The water-soluble Zn fraction is immediately available for plant uptake, while the exchangeable fraction, loosely bound to soil colloids, can be replaced by cations like Ca²z, Mg²z, or Kz (Liu *et al.*, 2020). Higher WS-Ex Zn under $ZnSO_4$ arises from its high solubility, releasing Zn²z ions that adsorb onto negatively charged colloids (Zhang *et al.*, 2014). This weak binding enables dynamic exchange between soluble and exchangeable pools (Sarangthem *et al.*, 2019). WS-Ex Zn peaked at 45 days due to rapid solubilization and adsorption, then declined as Zn became immobilized by adsorption, precipitation, and microbial complexation-typical of soil Zn dynamics. Similar results were reported Zhang *et al.*

Table 1. Zn–sequential fractionation procedure:

S. No.	Fractions	Solution(ml)	Soil (g):Solution(ml)	Condition
1	Water soluble–Zn(WS-Zn)	Distilled water	5:20	Shaken for one hour, centrifuge
2	Exchangeable – Zn (EX-Zn)	1N (Ammonium acetate) NH_4 OAC at pH 7.0	5:20	Shaken for one hour, centrifuge
3	Complexed – Zn (Comp-Zn)	0.05NCu (OAC)	5:20	Shaken for one hour, centrifuge
4	Zn associated with organic fraction (OB Zn)	H_2O_2(30%) Distilled water 0.05NCu (OAC)	5:20	Shaken for one hour, centrifuge
5	Occluded Zn and Zn bound by carbonates and other acid soluble minerals	HCL(0.1M) 20 ml	5:20	Shaken for one hour, centrifuge

(2014) and Kandali *et al.* (2016).

Complex zinc fractions (Comp-Zn)

Complex zinc fractions refer to zinc that is bound to organic matter, minerals, or precipitated as insoluble compounds. These fractions are considered more stable and immobile, serving as a long-term reservoir of zinc that is less available for plant uptake. Complexed Zn fraction recorded the second lowest value next to WSEx-Zn in terms of concentration. The periodic results of the complex zinc (Comp-Zn) fraction throughout the incubation period, up to 90 days, are illustrated in Fig. 2. The application of zinc sulfate ($ZnSO_4$) (T_2) significantly increased the Comp-Zn fraction up to the 45th day, reaching its highest peak of 11.7 mg kg^{-1} at that time, followed closely by zinc lysinate. Comp-Zn levels peaked at 45 days in all treatments then declined significantly. Zinc binds to soil colloids (clay, Fe and Mn oxides, and other inorganic surfaces) and complexes with organic matter or precipitates as Zn compounds, acting as a reservoir (Zhou *et al*., 2018). The results are in conformity with that of Baishya (2018) and Bairagi *et al*. (2020).

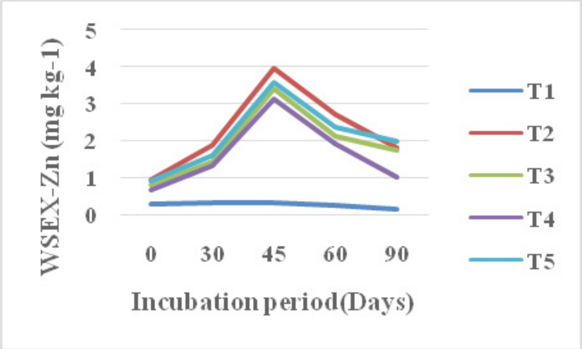

Fig. 1. Effect of zinc sources on the periodical changes of Water soluble plus exchangeable zinc fractions (WSEX-Zn) in clay loam soil

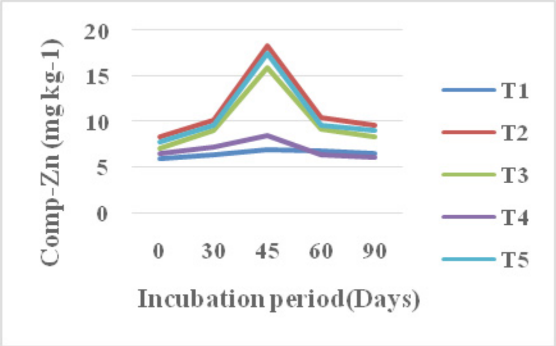

Fig. 2. Effect of zinc sources on the periodical changes of Complexed zinc fractions (Comp-Zn) in clay loam soil

Organically bound Zn

Data on organically bound zinc are presented in Fig. 3. The amount of zinc in the organically bound form exhibited an increase following the application of zinc sulfate ($ZnSO_4$) (T_2) immediately after submergence, peaking at 45 days (15.81 mg kg^{-1}) of submergence, followed by zinc lysinate. After this peak, all treatments showed a declining trend.

Organically bound Zn forms through soil organic matter, mainly humic and fulvic acids with functional groups (-OH, -COOH, -SH, -C=O) that strongly bind Zn. Its rise until 45 DAI is due to Zn release from sesquioxides under reduction and chelation by decomposition products (Prasad and Sarangathem, 1993). The later decline may stem from instability of Zn-organic complexes at low redox potential (Patnaik *et al.*, 2011). Initially, only 1.7–3.6% of applied Zn was organic, but this fraction increased during incubation. Similar observations

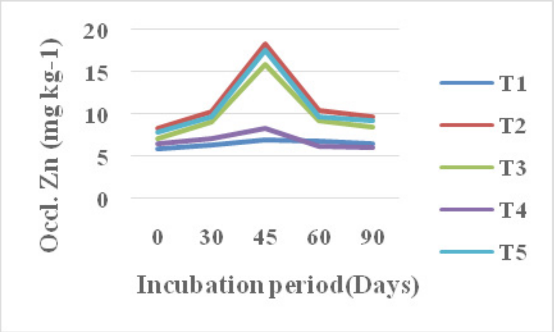

Fig. 3. Effect of zinc sources on the periodical changes of organic bound zinc fractions (OB Zn) in clay loam soil

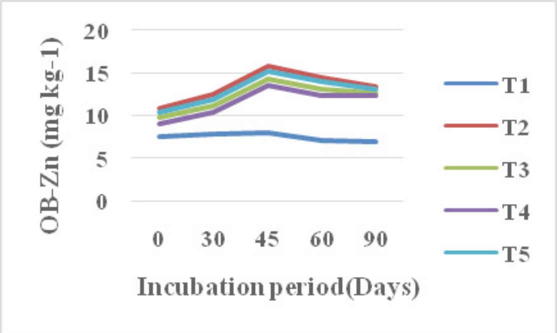

Fig. 4. Effect of zinc sources on theperiodical changes of Occluded zinc fractions (Occl. Zn) in clay loam soil

have also been reported by Dhaliwal and Dhaliwal (2019) and Mondal *et al.* (2024).

Occluded zinc fractions

A significantly higher content of occluded zinc (18.3 mg kg^{-1}) was recorded at 45 days after incubation (DAI) in the treatment receiving zinc sulfate ($ZnSO_4$) (T_2), compared to the lowest level (5.9 mg kg^{-1}) found in the control treatment at 1 DAI throughout the incubation period (Fig. 4). The application of $ZnSO_4$ (T_2) also exhibited the highest percentage contribution of occluded zinc to the total zinc content. The peak level of occluded zinc was achieved at 45 days of incubation for the $ZnSO_4$ (T_2) treatment, after which there was a significant decline across all treatments (Fig. 4). The increase in occluded Zn with higher Zn application is due to fertilizer addition and its transformation through interaction with amorphous sesquioxides (Talukdar *et al.*, 2013). Occluded Zn, immobilized within soil particles via organic matter complexation, is less available to plants. Its higher content compared to WS-Ex Zn and Comp-Zn may be attributed to the strong adsorption capacity of amorphous sesquioxides, owing to their large surface area, as noted by Kandali *et al.* (2016).

Residual-Zn (Res-Zn)

Residual zinc refers to the amount of zinc retained in weathered parent materials, constituting approximately 80% of the total zinc in soil. A higher percentage of zinc in the residual fraction in many soils indicates its greater tendency to become unavailable to plants (Fig. 5). The residual Zn fraction peaked at 45 DAI across all treatments, and then declined. At 45 DAI, the highest residual Zn (53 mg kg^{-1}) was observed with $ZnSO_4$, followed by zinc lysinate (49 mg kg^{-1}), while the control recorded the lowest (42 mg kg^{-1}). Zn fraction distributions were significantly influenced by the Zn source. At 90 DAI, the order of fractions was WS-Ex < complexed < organic-bound < occluded < residual. Similar high residual Zn fractions have been reported by Baishya (2018) and Mondal *et al*. (2024)

Residual Zn, a major part of total Zn, represents labile-to-non-labile conversion. Predominantly lithogenic and bound to mineral structures, it increased up to 45 DAI, with prolonged submergence enhancing Zn immobilization. Similar observations were reported by Kandali *et al.* (2016), and Ilavarasi *et al.* (2019). Residual Zn is mostly embedded in the soil matrix, remaining unavailable except under extreme conditions (Ma and Rao, 1997).

Total Zinc (Total-Zn)

Total Zn (total-Zn) reflects overall Zn accumulation in soil, influenced by parent material (Fig. 6), and is calculated as the sum of WS-Zn, Ex-Zn, OB-Zn, Comp-Zn, Res-Zn, and other fractions. The highest total-Zn (107.9 mg kg^{-1}) was recorded with $ZnSO_4$ at 45 DAI, while the control had the lowest (5.97 mg kg^{-1} at 1 DAI). Total Zn peaked at 45 DAI, then declined, as applied Zn solubilizes and transforms into various fractions. $ZnSO_4$ produced the highest total Zn due to its rapid solubility, whereas other sources released Zn more slowly. Overall, $ZnSO_4$ provided a faster and more sustained increase in soil Zn. These findings align with Bairagi *et al*. (2020) and Liu *et al*. (2020).

The correlation analysis showed that total zinc showed a positive correlated with all zinc fractions at 45 DAI (Table 2). The coefficient of correlation ranged between 0.837**to 0.976**. It was in descending order: Occl-Zn (r = 0.976**)> Res Zn (r= 945**) >Comp -Zn (r = 0.902**)> OB-Zn(r = 878**)> WSEx-Zn(r = 0.838**)

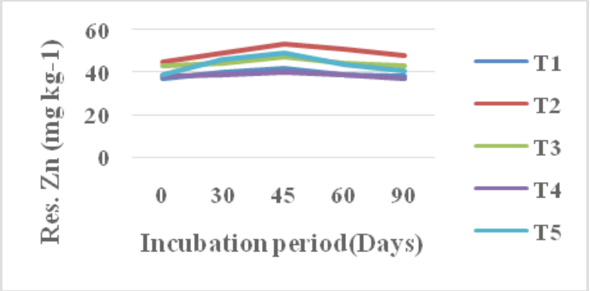

Fig. 5. Effect of zinc sources on the periodical changes of Residual zinc (Res. Zn) in clay loam soil

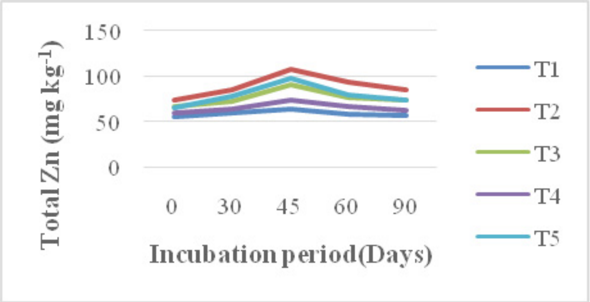

Fig. 6. Effect of zinc sources on the periodical changes of Total zinc (Tot. Zn) in clay loam soil

Release behaviour of DTPA-extractable zinc in soil

The incubation study assessed Zn release from $ZnSO_4$, Zn HEDP, ZnO_4, and Zn lysinate as DTPA-extractable Zn in control and amended soils (Fig. 7). Release was slow during the first 30 days, peaked at 45 DAI, then declined. $ZnSO_4$ (5 kg ha^{-1}) showed the highest release, followed by Zn lysinate, in the order: $ZnSO_4$ > Zn lysinate > Zn HEDP > ZnO > Control. On day one, $ZnSO_4$ released 2.5 mg kg^{-1}, peaked at 2.72 mg kg^{-1} at 45 DAI, then fell to 1.81 mg kg^{-1} by day 90; Zn lysinate reached 2.23 mg kg^{-1} at 45 DAI. Potentially available Zn declined in all treatments (Imran *et al*., 2021) with the control lowest (0.39 mg kg^{-1} at day 90).

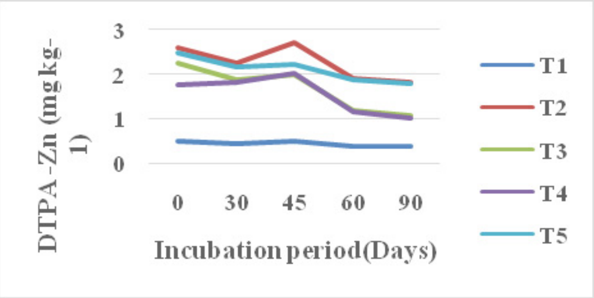

Fig. 7. Effect of zinc sources on zinc release pattern in Clay Loam Soil

$ZnSO_4$ released the most Zn during incubation due to its high solubility, rapidly dissociating into Zn^{2}z ions more available than less soluble sources like ZnO or complexes. DTPA-extractable Zn was highest with $ZnSO_4$, especially early, as it faced limited fixation onto soil colloids (Nawaz *et al*., 2015). Availability peaked at 45 DAI, then declined, but remained higher than Zn HEDP, Zn lysinate, or ZnO (Bairagi *et al*., 2020; Imran *et al*., 2021). DTPA-Zn was strongly correlated with WS-Ex Zn, Occl-Zn, Comp-Zn, OB-Zn, and Res-Zn at 45 DAI. The coefficient of correlation ranged between 0.871** and 0.985**. It was in descending order: OB-Zn (r=0.985**)> WSEX-Zn(r=0.983**)> Occl-Zn > (r=0.898**)>Comp-Zn (r=0.895**)>Res-Zn (r=0.872**).

Soil pH and Electrical Conductivity

Zinc source treatments significantly affected soil pH (Figs. 8–9). $ZnSO_4$ caused the greatest pH reduction (7.00), while Zn-HEDP showed the highest pH (7.28), indicating minimal change. These results align with Ayyar *et al*. (2019). Zinc release is influenced by clay type, micronutrient status, wet–dry cycles, pH, and moisture. Early nutrient uptake can also affect plant nutrient concentrations (Ma and

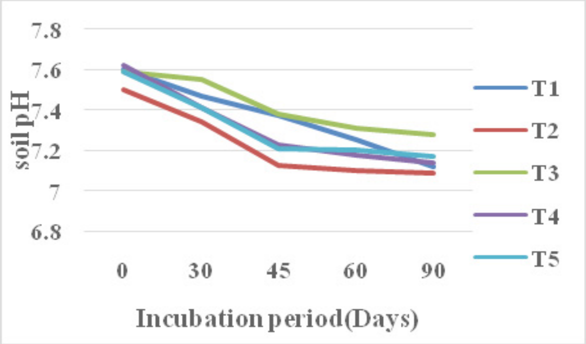

Fig. 8. Effect of zinc sources on on the periodical changes of pH in clay loam soil

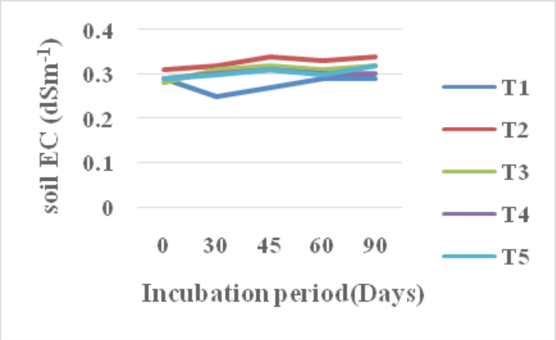

Fig. 9. Effect of zinc sources on on the periodical changes of EC in clay loam soil

Table 2. The correlation coefficient between DTPA-Zn, Total-Zn and Soil pH with different zinc fractions as influenced of different sources of zinc at clay loam

	WSEX-ZN	Comp-Zn	OB-Zn	Occl-Zn	Res-Zn	Total -Zn	DTPA-Zn	pH
WSEX-ZN	1.000							
Comp-Zn	0.690**	1.000**						
OB-Zn	0.996**	0.729**	1.000					
Occl-Zn	0.794**	0.796**	0.839**	1.000				
Res-Zn	0.613**	0.895**	0.672**	0.934**	1.000			
Total -Zn	0.838**	0.902**	0.878**	0.976**	0.945**	1.000		
DTPA-Zn	0.983**	0.896**	0.985**	0.899**	0.872**	0.871**	1.000	
pH	-0.638*	-0.812*	-0.655*	-0.479*	-0.529*	-0.635*	-0.749*	1.000

Rao, 1997). The soil pH was highly significantly and negatively correlated with WSEx-Zn (r= -0.638*), Comp-Zn (r= -0.811*), Occl-Zn (r= -0.655*), OB-Zn (r= -0.529*) and Res-Zn (r= -0.634*) at 45 DAI which showed that increase in zinc values at all fractions showed a decrease in pH.

Zinc sources also affected soil EC. $ZnSO_4$ caused the highest EC increase, followed by Zn lysinate, while the control remained lowest. EC decreased at 30 DAI but increased at 45, 60, and 90 DAI for all treatments, consistent with Tripathi *et al*. (2017) and Ayyar *et al*. (2019).

Conclusion

The 90-day incubation study showed Zn fractions in clay loam soil ranked as WS-Ex Zn < complexed Zn < organically bound Zn < occluded Zn < residual Zn, with WS-Ex the least and residual the largest. All fractions peaked at 45 DAI, then declined, with $ZnSO_4$ most effective in releasing plant-available WS-Ex Zn during the first 45 days

Author's contribution

Conceptualization of research work and designing of experiments (SR, MT, PP, SS, KS); Execution of field/lab experiments and data collection (SR); Analysis of data and interpretation (SR, MT, PP, SS, KS); Preparation of manuscript (SR, MT, PP, SS, KS)

Declaration

The authors should declare that they do not have any conflict of interest.

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