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Comparative efficacy of individually and combined application of compost, biochar, and bentonite on Ni dynamics in a calcareous soil

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Abstract. In Iran, a considerable proportion of agricultural soils are contaminated with various heavy metals (HMs), including nickel (Ni), necessitating remediation to mitigate their transfer into the food chain. However, there remains a scarcity of research on the effectiveness of applying organic and inorganic materials, either individually or in combination, for Ni immobilization in contaminated calcareous soils. To address this gap, an incubation experiment as completely randomized design with three replications was conducted to compare the effect of different soil amendments, either individually or combined (municipal solid waste compost (M), bentonite (B), municipal solid waste compost biochar (MB), M+B, MB+B, MB+M each applied at 2% wt.) on Ni immobilization in a calcareous soil with three Ni contamination levels (0 (Ni_0), 150 mg kg^{-1} (Ni_1) and 300 mg kg^{-1} (Ni_2). The study employed analytical techniques such as SEM-EDX, XRD, FTIR, sequential extraction, and DTPA-release kinetics to assess the efficiency of these amendments on stabilizing Ni in the soil. Elevating Ni levels from Ni_0 to Ni_2 increased Ni concentrations across all soil fractions, especially in Fe/Mn oxides (FeMnOx) and organic matter (OM). All amendments except M enhanced Ni immobilization by converting more labile fractions (WsEx, Car, FeMnOx) into residual (Res) form. While combined amendments were not more effective than single treatments, MB was the most efficient in stabilizing Ni. MB also exhibited the lowest 'a' and highest 'b' values attributed to the power function kinetics model, indicating superior Ni desorption reduction. These finding are likely due to its alkaline pH, ash content, and phosphorus content, which facilitate Ni precipitation. In contrast, M increased Ni desorption by raising its bioavailability (WsEx and Car fractions). The combined application of biochar (MB) with either bentonite (B) or compost (M) did not exhibit synergistic effects on the immobilization of Ni in the soil. In conclusion, the independent application of municipal solid waste-derived biochar appears to be a potentially effective amendment for enhancing Ni immobilization in contaminated calcareous soils.

1 Introduction

Heavy metal(loid)s (HMs) are toxic elements that can build up in farmland due to human activities like urbanization, industry, mining, and agrochemical use. Once in the soil, HMs are absorbed by crops, contaminating food and posing serious health risks. They also damage beneficial soil microbes, disrupting nutrient cycles and reducing agricultural output (Jahandari and Abbasnejad, 2024; Faraji et al., 2023; Munir et al., 2021). Nickel (Ni) is a unique HMs that, unlike mercury (Hg), cadmium (Cd), and lead (Pb), is essential for plant growth in minute quantities.

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However, at concentrations exceeding 35 mg kg^{-1} soil, Ni becomes harmful, causing physiological and morphological disruptions that severely impair plant development (Shahzad et al., 2018). In Iran, industrial-adjacent agricultural soils exhibit alarmingly high Ni levels, with an average concentration of 350 mg kg^{-1}, far exceeding environmental safety thresholds (Shahbazi et al., 2022). A nationwide study analyzing 711 soil samples revealed Ni concentrations ranging from 2.79 to 770 mg kg^{-1}, with 11.3% of soils surpassing permissible limits (Shahbazi et al., 2020).

When direct removal of toxic ions is unfeasible, immobilizing them on solid surfaces is the optimal approach (Derakhshan Nejad et al., 2018). To achieve this, materials such as biochar, clay minerals, and bio/polymers are incorporated into soils. These additives, known as "soil modifiers," significantly influence the soil's physicochemical properties, sorption capacity, and microbial communities (Jin et al., 2024; Gong et al., 2024; Rasheed and Moghal, 2024). Compost improves HMs immobilization through increased soil organic matter (SOM), which enhances metal complexation and cation exchange capacity (Asemoloye et al., 2020). However, its efficacy varies with decomposition rates and may be limited by potential HMs release during compost mineralization (Hobbs et al., 2011). Biochar, a carbon-rich material produced through biomass pyrolysis, exhibits strong sorption properties due to its large surface area, porous structure, abundance of surface functional groups (e.g., carboxyl, hydroxyl, and phenolic groups), and high aromaticity (Comath et al., 2025; Afshar and Mofatteh, 2024). Its effectiveness in immobilizing HMs in soil depends on factors such as the original feedstock, pyrolysis temperature, particle size, and soil type (Gholizadeh et al., 2024; Fakhar et al., 2025). On the other hand, natural bentonite primarily consists of montmorillonite, a 2:1 clay mineral known for its high cation exchange capacity (CEC) and excellent water retention properties (Zhang et al., 2020). Bentonite clay effectively eliminates HMs from soil, offering a solution due to its permanent surface charges and isomorphic substitution properties (Mi et al., 2020; Xie et al., 2018). Also, from an economic standpoint, bentonite is a highly attractive soil amendment due to its affordability and widespread global availability (Peng and Sun, 2012).

It is widely recognized that the toxicity of HMs is determined by their geochemical fractions rather than their total concentrations in soil (Pelfrene et al., 2020). Additionally, accurately assessing the health risks posed by HMs in contaminated soils relies on how precisely plant uptake is simulated (Krauße et al., 2019). Therefore, integrating sequential extraction methods with HMs release kinetics studies is essential for gaining deeper insights into the behavior of these metals in polluted soils (Wang et al., 2021). Recent studies have commonly employed time-dependent release kinetic experiments using single extractants, along with sequential extraction procedures, to assess how effectively soil amendments immobilize HMs in contaminated soils (Boostani et al., 2024a; Boostani et al., 2023). Analysis of a contaminated soil sample demonstrated that Ni and Cu were predominantly present in residual (Res) fractions (53% and 57%, respectively), with subsequent partitioning in organic (OM), Fe-Mn oxide (FeMnOx), carbonate (Car), and soluble +exchangeable (WsEx) fractions. The application of bentonite significantly altered metal speciation, reducing labile (WsEx) fraction while enhancing retention in FeMnOx and Res phases. This shift effectively decreased the mobility and bioavailability of the HMs, highlighting bentonite's potential as an immobilizing agent in remediation strategies (Gao and Li, 2022a). In another study, Boostani et al. (2023) investigated biochar (cow manure, municipal compost,

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licorice root pulp; 300/600 °C) effects on Ni stabilization in a Ni-contaminated calcareous soil. They indicated that High-temperature biochars (600 °C) reduced mobile Ni (10–42%) and increased Res Ni (13–38%), with ash-rich biochars most effective.

So far, research about comparing the efficacy of individually and combined application of compost, biochar, and bentonite on chemical fractions and release kinetics of Ni in contaminated calcareous soils has been limited, leaving the effectiveness of this approach uncertain. We hypothesized that (1) biochar will outperform compost and bentonite alone due to its stable carbon structure and alkalinity, and (2) combined applications will enhance Ni immobilization through complementary mechanisms. Therefore, this study aimed to address the knowledge gap by evaluating whether combining these three materials (compost, biochar, and bentonite) for soil Ni immobilization is scientifically supported. Moreover, the research aimed to uncover the fundamental soil chemical mechanisms driving these observed effects.

2 Materials and methods

2.1 Soil Collection, Characterization, and Ni Contamination

A topsoil sample (0-30 cm) was collected from a calcareous soil in Darab, southern Iran, using an auger. The soil was air-dried immediately, sieved through a 2 mm mesh, and stored at room temperature for further physical and chemical analyses. Standard laboratory methods (Sparks et al., 2020) were employed for soil characterization. For Ni contamination, 2 kg of soil was placed in plastic containers and mixed with 500 mL of a $NiCl_2$ solution to achieve a final concentration of 150 and 300 mg Ni kg^{-1} soil. The treated soil was then air-dried at ambient temperature, brought to field capacity (FC) using distilled water, and dried again. This wetting-drying cycle was repeated three times to ensure Ni equilibration, simulating natural field conditions (Boostani et al., 2024a).

2.2 Biochar preparation and its characteristics

Municipal solid waste compost was chosen as feedstock for biochar production. It was first air-dried and finely ground. The processed biomass was then oven-dried at 105 °C for 24 hours. Slow pyrolysis was carried out in an electric muffle furnace (Shimifan, F47) under limited oxygen conditions (Khalili et al., 2024). The temperature was gradually increased at a rate of 5 °C per minute from room temperature until reaching 500 °C, which was maintained for two hours. After pyrolysis, the biochars were allowed to cool naturally and then sieved through a 0.5 mm mesh to ensure consistent particle size. The chemical properties of the biochars were assessed using standard laboratory methods (Singh et al., 2017). Fourier Transform Infrared (FTIR) spectroscopy (Shimadzu DR-8001) with the KBr pellet technique was used to analyze surface functional groups. Additionally, the surface morphology of the biochars was examined using scanning electron microscopy—Energy Dispersive X-ray Spectroscopy (SEM-EDX) (TESCAN-Vega3, Czech Republic). X-ray diffraction (XRD) analysis was performed using a GNR XRD Explorer diffractometer (Italy) with Cu Kα radiation (λ = 1.54178 Å), scanning a 2θ range of 10° to 70°.

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2.3 Preparation and composition of bentonite

The bentonite used in this research was sourced from natural mines in Semnan province, Iran. The raw material was crushed and sieved to achieve a particle size below 0.5 mm. Table 1 presents the elemental analysis of the bentonite powder employed in the study.

Table 1. Elemental composition of the natural bentonite (%).

Cl	TiO_2	MgO	CaO	K_2O	Na_2O	Fe_2O_3	Al_2O_3	SiO_2
0.48	0.2	2.68	0.39	0.49	2.11	1.64	11.93	70.56

2.4 Experimental Design and Incubation Procedure

The experiment followed a factorial arrangement in a completely randomized design with three replicates. The treatments consisted of three Ni levels (0 (Ni_0), 150 (Ni_1), and 300 (Ni_2) mg kg^{-1} soil supplied as $NiCl_2$) and Seven soil amendment types: control (no amendment, Cl), municipal solid waste compost (M), bentonite (B), municipal solid waste compost biochar (MB), M+B, MB+B and MB+M). Each amendment was applied at a 2% (w/w) rate. 200 g of Contaminated soil samples (spiked with 150 and 300 mg Ni kg^{-1}) was placed in plastic containers and thoroughly mixed with the designated amendments. The soil moisture was adjusted to near field capacity using distilled water. The samples were incubated at 25±2°C for 90 days, with moisture levels maintained by daily additions of distilled water. After incubation, the soil was air-dried, sieved (2 mm), and stored for subsequent chemical analysis.

2.5 Fractionation of soil Ni

Nickel in the soil was partitioned into five chemical fractions using the sequential extraction procedure described by Salbu and Krekling (1998). The fractions included: water-soluble + exchangeable (WsEx), carbonate-bound (Car), iron-manganese oxide-bound (FeMnOx), organic matter-bound (OM) and residual (Res). The residual fraction was determined by subtracting the sum of the first four fractions from the total soil Ni content. Total Ni concentration in the soil was measured following the method of Sposito et al. (1982).

2.6 Nickel desorption study

The Ni desorption experiment was conducted by mixing 10 g of air-dried soil with 20 mL of DTPA solution (pH 7.3) (Lindsay and Norvell, 1978). The extraction process was performed using an end-over-end shaker at 25 ± 2°C for varying time intervals (5, 15, 30, 60, 120, 360, 720, and 1440 minutes). Following each extraction period, samples were centrifuged at 4000 rpm for 10 minutes. The resulting supernatants were filtered through Whatman No. 42 filter paper, and Ni concentrations were determined by atomic absorption spectroscopy (AAS; PG 990, PG Instruments Ltd., UK). The time-dependent Ni release (q_t) was modeled using a power function kinetic equation ($q_t = at^b$). Model validity was assessed based on high coefficient of determination (r^2) values and low standard error of estimate (SEE).

2.7 Statistical Analysis

All data were processed using MSTATC software. Treatment means were compared using Tukey's multiple range test at $p \leq 0.05$. Graphical representations were prepared in Microsoft Excel 2013, while Pearson correlation analyses were performed with SPSS 22.0 software.

3 Results and discussions

3.1 Soil characteristics

Table 2 summarizes the pre-contamination physicochemical properties of the soil, characterized by a sandy loam textural class. The soil was slightly alkaline, with elevated calcium carbonate (42.0%), indicating potential phosphorus fixation and reduced micronutrient availability. Low electrical conductivity (0.58 dS m^{-1}) confirms negligible salinity, while minimal organic carbon (0.39%) suggests restricted nutrient cycling. These attributes are consistent with the soil characteristics found in the arid and semi-arid regions of Iran (Mirkhani et al., 2010). Although total Ni (38.0 mg kg^{-1}) was slightly higher than the typical background level (35 mg kg^{-1}) (Shahzad et al., 2018), its low DTPA-extractable fraction (0.20 mg kg^{-1}) indicates limited bioavailability under prevailing conditions.

Table 2. Some physicochemical characteristics of the soil sample prior to contamination.

characteristics	value
Sand (%)	53.7
Silt (%)	24.3
Clay (%)	22.0
Soil textural class	Sandy loam
pH_s	7.50
Electrical conductivity (dS m^{-1})	0.58
Soil organic carbon (%)	0.39
Cation exchange capacity ($cmol_c$ kg^{-1})	12.0
Calcium carbonate equivalent (%)	42.0
Ni-DTPA (mg kg^{-1})	0.20
Total Ni (mg kg^{-1})	38.0

3.2 Chemical properties of the soil amendments

Table 3 shows the chemical properties of three soil amendments with varying physicochemical properties. The MB had the highest electrical conductivity (2.26 dS m^{-1}) and pH (9.12) among the amendments, followed by M (1.84 dS m^{-1}, 7.42) and B (0.83 dS m^{-1}, 8.40). Organic-rich amendments (MB and M) contain quantifiable amounts of carbon (23.0%, 18.1%), nitrogen (1.85%, 2.50%), and phosphorus (0.42%, 0.29%), but B does not contain these nutrients. The MB is also significantly greater in ash content (65.0%) and Ni concentration (21.6 mg kg^{-1}) compared to the M (50.0%, 14.0 mg kg^{-1}), reflecting its pyrolyzed nature. The H:C mole ratio, a measure of aromaticity (Mccall et al., 2024), is lower in the MB than in M, reflecting greater carbon stability in the biochar (Table 3). These contrasts emphasize the alkaline, nutrient-rich, and recalcitrant nature of MB relative to the greater nitrogen content of M and the more inert, mineralogical nature of B.

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Table 3. Chemical characteristics of applied amendments.

Characteristics	M	MB	B
EC (1:20) (dS m^{-1})	1.84	2.26	0.83
pH (1:20)	7.42	9.12	8.40
Total C (%)	18.1	23.0	--
Total H (%)	3.40	2.44	--
Total N (%)	2.50	1.85	--
Total P (%)	0.29	0.42	--
Total Ni (mg kg^{-1})	14.0	21.6	Nd
Ash Content (%)	50.0	65.0	--
*O+S content (%)	23.0	7.71	--
H:C mole ratio	2.25	1.27	--

Notes: M, Municipal solid waste compost; MB, Municipal solid waste compost biochar; B, Bentonite clay; *Determined by subtraction of ash, moisture, C, N and H from total mass

3.3 FTIR, SEM-EDX and XRD of the soil amendments

FTIR spectrum of MB, produced through pyrolysis of compost, exhibits several changes as a result of thermal degradation during biochar production (Figure 1). Most notably, decreased intensity or absence of C=O stretching vibrations near 1700 cm^{-1} (carboxyl groups) (Keiluweit et al., 2010) in the MB compared to M suggests a loss in CEC, which is corroborated by the lower O+S content of MB (Table 3). The intense band at 1432 cm^{-1} in MB may suggest greater contents of $CaCO_3$ or lignin-derived structures (Myszka et al., 2019) compared to M. Conversely, the weakened band at 1100 cm^{-1} specific for cellulose (Keiluweit et al., 2010) in MB suggests thermal degradation of the labile organic constituents by pyrolysis, once again pointing toward the structural transformation from compost to biochar. The FTIR spectrum of MB also shows characteristic mineral-associated vibrations, with Si-H stretching bands at 470 cm^{-1} and 800 cm^{-1} easily recognizable (Zemnukhova et al., 2015). The spectral features likely indicate the formation of organo-mineral complexes as a result of thermal transformation processes during pyrolysis (Lehmann and Joseph, 2024).

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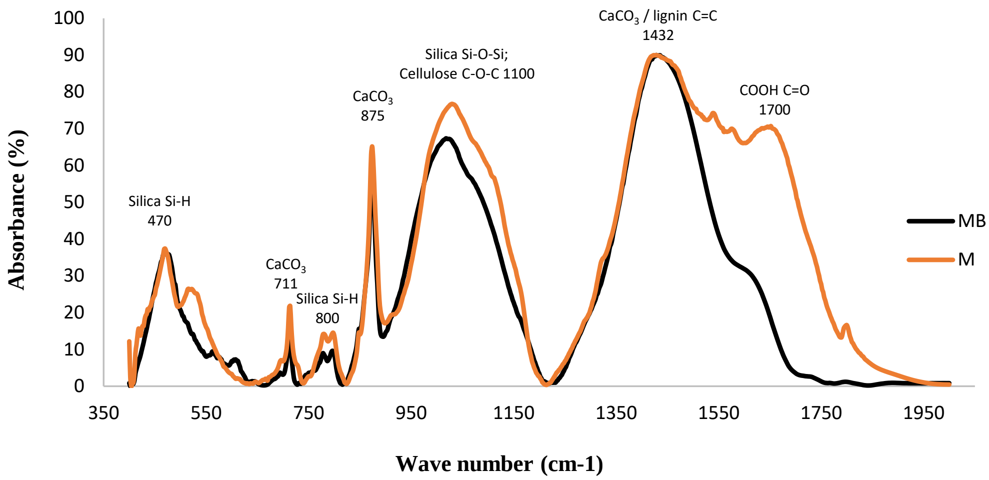

Figure 1. FTIR spectra for the organic amendments (M and MB) in the range of 400-2000 cm^{-1}
Notes: M, Municipal solid waste compost; MB, Municipal solid waste compost biochar

The EDX analysis showed distinct elemental composition for each amendment, in agreement with their respective organic or inorganic sources (Figure 2). Bentonite clay contained predominantly silicon (Si, 33.9%) and aluminum (Al, 7.10%), in agreement with its aluminosilicate mineral nature. Trace levels of elements such as iron (Fe, 1.99%) and alkali metals (Na and K) reflect the presence of natural impurities. On the other hand, the organic amendments contained significantly higher carbon (C, 19.9% for M; 26.1% for MB) and nitrogen (N, 4.22% for M; 3.50% for MB) content, which is a reflection of their compost-derived organic matter. MB had higher carbon content due to higher carbonization from the pyrolysis process. In addition, MB contained a significantly higher proportion of calcium (Ca, 22.2%), possibly originating from mineral additives or ash materials developed during the production of biochar.

The SEM images also presented surface structural differences in the amendments (Figure 2). Bentonite clay showed a close, laminated structure with level, platelet-shaped particles characteristic of aluminosilicate minerals, in line with its high Si and Al percentages. The M possessed a heterogeneous, porous structure with irregularly sized organic pieces, consistent with its organic origin and partially decomposed biomass. In contrast, the biochar (MB) possessed an extremely porous, broken morphology with a honeycomb structure, the outcome of pyrolytic treatment that maximizes surface area and promotes carbon retention.

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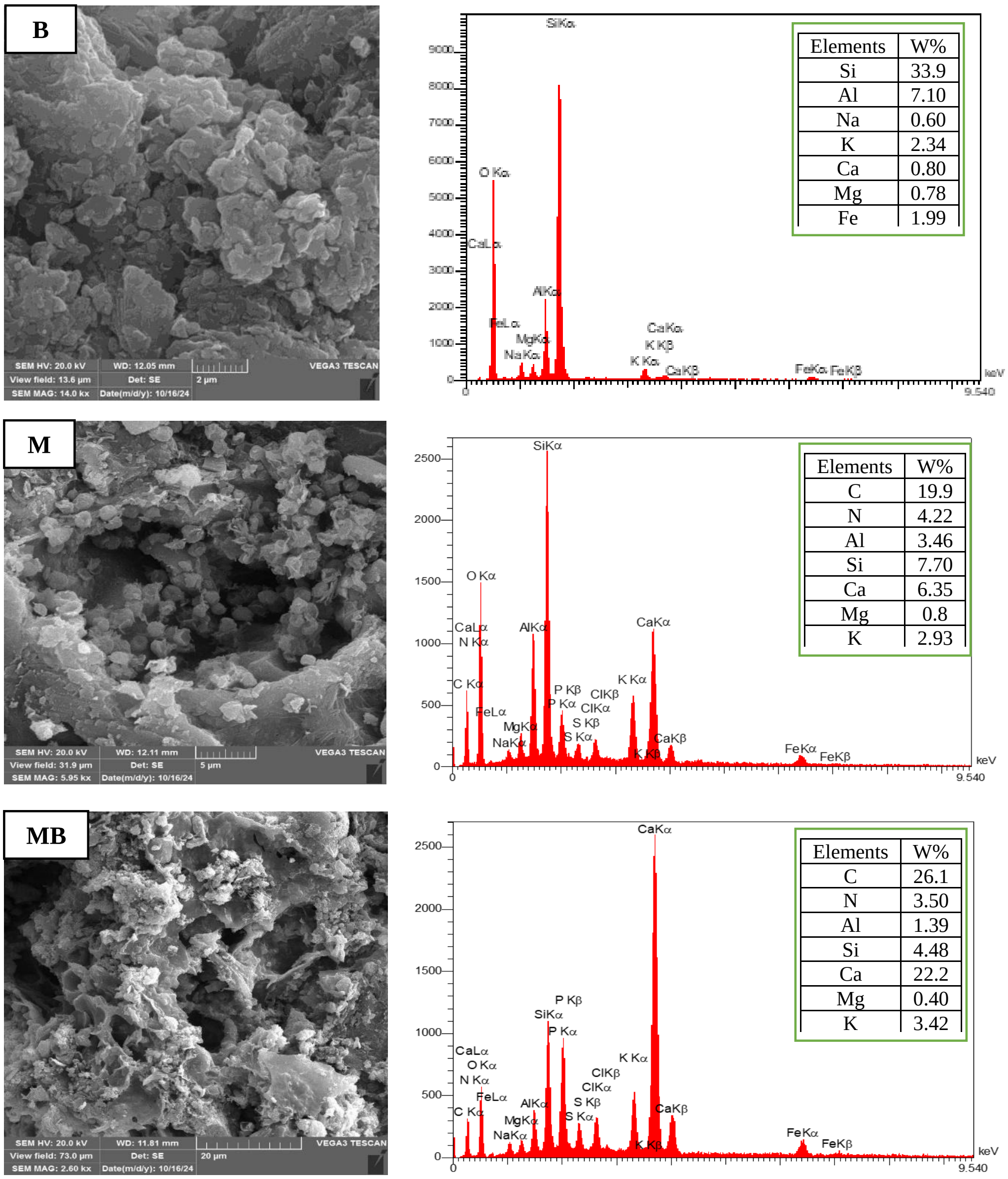

Figure 2. SEM-EDX of the organic and inorganic amendments (M, MB and B).
Notes: M, Municipal solid waste compost; MB, Municipal solid waste compost biochar; B, Bentonite clay.

The XRD analysis of the soil amendments contained distinct crystalline phases: the M exhibited a broad hump (2θ ~20–35°) typical for amorphous organic matter (e.g., cellulose, lignin) (Noorshamsiana et al., 2020) with quartz peaks (2θ ~26.6°) (Zuo et al., 2016) and minor traces of

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calcite (minor peaks at ~40-50°) (Al-Jaroudi et al., 2007). The MB showed sharp graphitic carbon peaks (2θ ~ 50°) (Destyorini et al., 2021) due to pyrolysis-induced crystallinity, along with persistent quartz (2θ ~26.6°) and potential calcium oxalate or phosphate (Peaks near 30°) phases (Petrova et al., 2019). Bentonite clay was dominated by montmorillonite reflections (2θ: 19.8°, 35°, and 62°), with quartz impurities (2θ: 21.8°, 26.6°, 35.9°) (Ikhtiyarova et al., 2012).

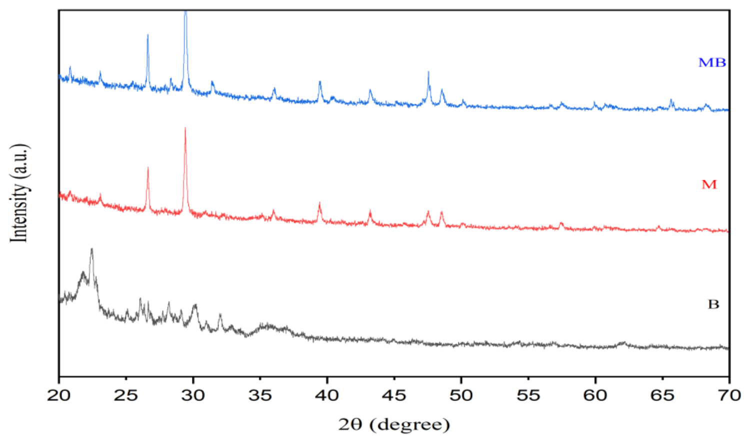

Figure 3. The XRD patterns for all the soil amendments (M, MB and B).
Notes: M, Municipal solid waste compost; MB, Municipal solid waste compost biochar; B, Bentonite clay.

3.4 Changing the soil pH as affected by different amendments

The influence of Ni levels, soil amendments, and their interactions on soil pH was statistically significant ($P < 0.01$). Elevating Ni levels from Ni_0 to Ni_2 resulted in a 0.03 unit decrease in soil pH, whereas no significant difference was observed between Ni_0 and Ni_1 levels (Table 4). This pH reduction at higher Ni concentrations may be attributed to Ni-induced displacement of exchangeable H^+ ions or the hydrolysis of Ni^{+2}, which releases protons into the soil solution (Sparks et al., 2022). Consistent with the present study, elevating Ni application from 150 to 300 mg Ni kg^{-1} in a calcareous soil contaminated with Ni resulted in an average reduction of 0.20 units in soil pH (Boostani et al., 2020). In contrast, amendments differentially increased soil pH, with the most pronounced effect (0.24 units) recorded for the MB+B treatment, while the minimal increase (0.05 units) was associated with M (Table 4). The pH-enhancing effect of amendments (M, MB, and B) can be ascribed to their liming properties, as confirmed by the FTIR (Figure 1), EDX (Figure 2) and chemical composition (Table 3) analysis. Both biochar and bentonite exhibit inherent alkalinity, consistent with their established role in mitigating soil acidity (Xie et al., 2024). The increase in soil pH observed with the B addition could be attributed to its high levels of SiO_2, MgO, and CaO (Table 1), which help neutralize soil acidity (Afzal et al., 2024). Additionally, biochar contributes to higher pH by exchanging its surface salt-based ions (Na^+, Ca^{2+}, K^+, and

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Mg^{2+}) with acidic H^+ in the soil, thereby boosting salt-based ion saturation and improving soil alkalinity (Lin et al., 2023). Notably, combined amendments like MB+B, M+B yielded higher soil pH values (7.61 and 7.57, respectively) than individual applications (M, B, MB) (Table 3), suggesting synergistic mechanisms wherein bentonite's high CEC may enhance retention of base cations derived from biochar or compost, thereby further neutralizing soil acidity. In agreement with our results, Xie et al. (2024) observed that a 1:1 weight ratio mixture of corn straw biochar and bentonite led to a greater rise in soil pH compared to their separate applications. Interaction analysis revealed the highest and lowest pH values in the Ni_0×MB+B (7.69) and Ni_0×Cl (7.21) treatments, respectively. Additionally, Ni application appeared to attenuate soil pH variability across amendments compared to the Ni_0 level (Table 4).

Table 4. Changing the soil pH as affected by the application of different amendments

	Cl	M	B	MB	M+B	MB+B	MB+M	
Ni_0	7.21^{j}	7.36^{i}	7.53^{de}	7.47^{fg}	7.63^{b}	7.69^{a}	7.60^{bc}	7.50^{A}
Ni_1	$7.49^{e\text{-}g}$	7.47^{fg}	7.54^{de}	7.47^{fg}	7.57^{cd}	7.54^{de}	7.48^{fg}	7.51^{A}
Ni_2	7.41^{h}	7.42^{h}	7.47^{fg}	7.42^{h}	7.51^{ef}	7.60^{bc}	7.45^{gh}	7.47^{B}
	7.37^{F}	7.42^{E}	7.51^{C}	7.45^{D}	7.57^{B}	7.61^{A}	7.51^{C}	

Notes: Cl, control (without amendment addition); M, municipal solid waste compost; B, bentonite clay; MB, municipal solid waste compost biochar; Ni_0, without Ni application; Ni_1, application of 150 mg Ni kg^{-1} soil; Ni_2, application of 300 mg Ni kg^{-1} soil. Numbers with same lower letters (interaction effects), in each section, had no significance difference to each other ($P < 0.05$). Also, numbers with same capital letters (main effects), in each section, had no significance difference to each other ($P < 0.05$).

3.5 Changes of soil Ni chemical fractions as affected by the amendment application

The Ni levels, soil amendment types, and their interactions exhibited statistically significant influences on Ni concentrations in the WsEx, Car and Res fractions (Table 5). Overall, elevating Ni levels from Ni_0 to Ni_2 resulted in significant increases in Ni concentrations across all chemical fractions, with the most pronounced rise observed in the FeMnOx and OM fractions (16.8-fold and 15.4-fold), respectively. In calcareous soils, the elevated pH (typically >7.5) enhances the formation of FeMn oxides/hydroxides with numerous reactive sites, which act as strong sinks for Ni via specific adsorption, co-precipitation, and surface complexation (Sparks et al., 2022; Rinklebe et al., 2016). Furthermore, Ni strongly complexes with organic ligands, particularly humic and fulvic acids, via carboxyl and phenolic functional groups, leading to its preferential retention in organic matter-bound fractions under aerobic conditions (Huang et al., 2023). The present findings align with prior research by Boostani et al. (2020), which identified Res, OM and FeMnOx fractions as the dominant chemical forms of Ni in a highly Ni-contaminated calcareous soil. Moreover, Mailakeba and Bk (2021) demonstrated that increasing the application rate of Ni (supplied as $NiCl_2$) from 0 to 180 mg kg^{-1} soil led to a substantial elevation in the soil Ni content within the OM fraction.

The efficacy of soil amendments in altering Ni concentrations within the WsEx fraction varied depending on initial soil Ni levels (Table 5). In the absence of added Ni (Ni_0), all amendments except M significantly reduced Ni-WsEx concentrations relative to the CL, with the most substantial decrease (60.7%) observed in the MB treatment (Table 5). Conversely, at the Ni_1 level, MB, MB+B, and MB+M treatments reduced Ni-WsEx concentrations, though only MB demonstrated statistically significant effect (Table 5). In contrast, at the Ni_2 level, amendments

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containing M (M, M+B, and MB+M) significantly increased Ni-WsEx content compared to the CL, whereas other amendments had no significant impact (Table 5). Overall, the application of M consistently led to an increase in Ni concentrations within the WsEx fraction across all the Ni levels (Table 5). Additionally, the effectiveness of combined amendments containing M, such as M+B and MB+M, in reducing Ni concentrations in the WsEx fraction appeared to be lower compared to the application of each component individually (B and MB) (Table 5). The municipal solid waste compost possesses a limited number of binding sites; however, its capacity to retain HMs is influenced by both the metal's affinity for the binding sites and the compost's pre-existing trace element load. While Pb exhibits strong binding affinity, Ni does not (Cao et al., 2023). The M contained considerable concentrations of Ni (Table 3) as well as other competing cations such as Ca, Mg, and K (Figure 2). Consequently, the available binding sites may become saturated, thereby reducing the compost's effectiveness in immobilizing newly introduced Ni. In other point of view, during the microbial decomposition process, compost may release dissolved organic carbon (DOC), which can chelate Ni and maintain it in soluble forms (Flury et al., 2015). Conversely, among all treatments, MB demonstrated the greatest efficacy in reducing Ni concentrations in the WsEx fraction under non-contaminated (Ni_0) and moderately contaminated (Ni_1) conditions. This result can likely be attributed to the alkaline properties of MB, characterized by its high pH, ash content, and phosphorus levels (Table 3), which promote the precipitation of Ni as hydroxides, carbonates, and phosphates in the soil (Boostani et al., 2024b). Nevertheless, at the highest contamination level (Ni_2), the application of MB did not significantly affect soil Ni content in the WsEx fraction (Table 5). Although the porous structure of MB (Figure 2) provides numerous adsorption sites for Ni^{+2} ions, these sites may become saturated under high Ni concentrations, resulting in the persistence of residual Ni within the WsEx fraction (Liang et al., 2021).

The experimental soil, despite its calcareous nature, exhibited unexpectedly low Ni concentrations in the Car fraction compared to the Res and FeMnOx fractions (Table 5 and Figure 4). This observation suggests that either the standard 1 M sodium acetate (pH 5) extraction method may be insufficiently aggressive to fully dissolve carbonate-bound Ni, or that Ni demonstrates only marginal chemical preference for carbonate association in such soil environments (Rajaie et al., 2008). Statistical analysis of interactions revealed that under Ni_0 conditions, none of the amendments, whether applied singly or in combination, significantly altered the Ni content in the Car fraction (Table 5). At moderate Ni contamination (Ni_1), only MB, B, and their combined application (MB+B) produced a statistically significant reduction in Ni concentration within the Car fraction relative to the control, with no notable differences observed among these treatments (Table 5). Boostani et al. (2020) demonstrated that incorporating various crop residue-derived biochars into a Ni-contaminated calcareous soil effectively decreased Ni concentration within the Car fraction. Furthermore, previous research supports the observations regarding the bentonite application, with Gao and Li (2022b) documenting dose-dependent reductions in Ni content in the Car fraction following bentonite application to contaminated soils. Parallel findings by Boostani et al. (2025) stablished an inverse relationship between bentonite application rates and Cd concentration in the Car fraction of a polluted calcareous soil. Conversely, under high Ni contamination (Ni_2), treatments involving M alone or in combination with B and MB (M+B, M+MB) led to a marked elevation in the Car fraction compared to the control. The most substantial

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increase (50.8%) was recorded for the sole application of M (Table 5). In contrast, MB, B, and their combined use (MB+B) exhibited no significant influence on Ni content in this fraction at the Ni_2 level (Table 5).

Table 5. Soil Ni content in fractions of WsEx, Car and Res as affected by the application of amendments

	Cl	M	B	MB	M+B	MB+B	MB+M	
				WsEx-Ni				
Ni_0	6.75^{e-g}	7.15^{e-g}	4.30^{h-j}	2.65^{j}	4.15^{h-j}	3.57^{ij}	3.65^{ij}	4.62^{C}
Ni_1	8.09^{d-f}	9.40^{cd}	8.32^{d-e}	5.69^{g-i}	10.50^{bc}	6.15^{f-h}	7.79^{d-f}	7.99^{B}
Ni_2	12.20^{b}	17.19^{a}	12.29^{b}	10.53^{bc}	16.57^{a}	10.53^{bc}	15.54^{a}	13.55^{A}
	9.01^{A}	11.25^{A}	8.30^{B}	6.29^{C}	10.40^{A}	6.81^{C}	8.99^{B}	
				Car-Ni				
Ni_0	4.75^{h}	3.89^{h}	3.69^{h}	3.77^{h}	3.83^{h}	3.82^{h}	3.79^{h}	3.93^{C}
Ni_1	18.51^{f}	19.25^{ef}	14.08^{g}	15.24^{g}	20.67^{d-f}	15.73^{g}	19.64^{ef}	17.59^{B}
Ni_2	21.28^{c-e}	32.09^{a}	22.98^{c}	22.89^{cd}	29.34^{b}	21.49^{c-e}	28.80^{b}	25.55^{A}
	14.85^{B}	18.41^{A}	13.58^{C}	13.97^{C}	17.94^{A}	13.68^{C}	17.41^{A}	
				Res-Ni				
Ni_0	15.81^{m}	31.10^{l}	20.34^{m}	42.76^{k}	33.86^{kl}	41.88^{k}	55.71^{j}	34.49^{C}
Ni_1	76.70^{i}	79.63^{i}	93.27^{eh}	100.6^{fg}	86.65^{hi}	102.6^{fg}	104.8^{f}	92.01^{B}
Ni_2	132.8^{d}	118.6^{e}	146.1^{c}	172.1^{a}	136.6^{cd}	168.6^{ab}	160.3^{b}	147.8^{A}
	74.96^{C}	76.46^{C}	86.55^{B}	105.2^{A}	85.72^{B}	104.3^{A}	106.9^{A}	

Notes: Cl, control (without amendment addition); M, municipal solid waste compost; B, bentonite clay; MB, municipal solid waste compost biochar; Ni_0, without Ni application; Ni_1, application of 150 mg Ni kg^{-1} soil; Ni_2, application of 300 mg Ni kg^{-1} soil; WsEx-Ni, water and soluble form of Ni; Car-Ni, carbonate-bound Ni; Res-Ni, residual Ni.
Numbers with same lower letters (interaction effects), in each section, had no significance difference to each other ($P < 0.05$). Also, numbers with same capital letters (main effects), in each section, had no significance difference to each other ($P < 0.05$).

The interaction between Ni levels and soil amendments did not demonstrate statistically significant effects ($P < 0.05$) on altering Ni concentrations in the FeMnOx and OM fractions, although the main effects of each factor were individually significant ($P < 0.05$). Among the amendments, only B, MB, and their combination (MB+B) significantly reduced soil Ni content in the FeMnOx fraction compared to the control, whereas other treatments showed no significant impact. Nevertheless, no statistically significant differences were observed among the M, MB, and MB+B treatments (Figure 4). A plausible explanation is that over time, a portion of the soil Ni present in the FeMnOx fraction may undergo enhanced transformation into the more stable form like Res, particularly under biochar and zeolite amendments (Ali et al., 2019). The application of M and M+MB treatments significantly increased the Ni concentration in the OM fraction relative to the control, though no statistically significant difference was observed between these two treatments (Figure 4). The obtained result suggests that Ni ions preferentially form complexes with carboxyl functional groups on the M surface (as evidenced by C=O stretching vibrations at ~1700 cm^{-1} in Figure 1), demonstrating stronger binding affinity compared to other passivation agents (Bashir et al., 2018). The application of biochar and compost amendments elevates soil organic matter content, thereby improving its capacity for immobilizing both HMs and organic contaminants through enhanced adsorption mechanisms (Li et al., 2020; Puga et al., 2015). Previous studies have documented that biochar and compost amendments can significantly increase the proportion of soil HMs associated with the OM fraction (Li et al., 2021; Paradelo et al., 2018).

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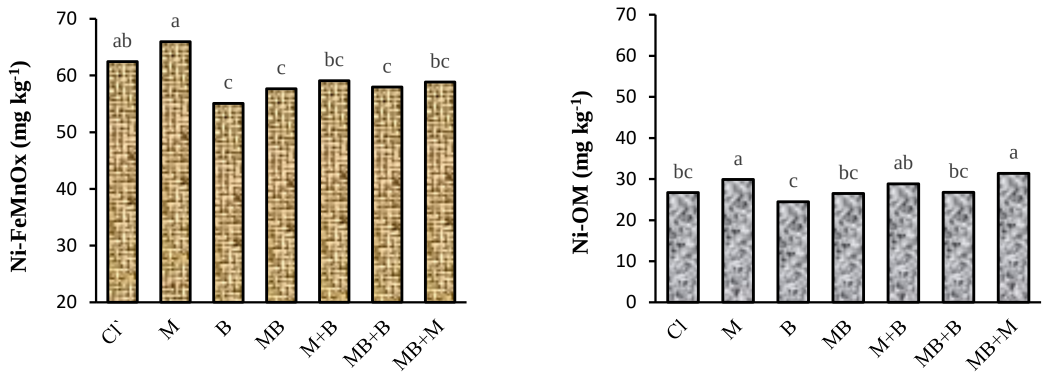

Figure 4. The main effects of amendments on changing the soil Ni concentration in the FeMnOx and OM fractions. Notes Cl, control (without amendment addition); M, municipal solid waste compost; B, bentonite clay; MB, municipal solid waste compost biochar. Ni-FeMnOx, Ni bound to iron-manganese oxides; Ni-OM, Ni bound to organic matter. Numbers with same lower letters had no significance difference to each other ($P < 0.05$).

The Res fraction represents the most stable chemical form of HMs in soil, bound within the lattice structure of clay minerals and exhibiting no bioavailability (Shen et al., 2022). At the Ni_0 level, the application of all amendments significantly elevated the Ni concentration in the Res fraction compared to the control, with the greatest increase observed under the combined MB+M treatment (3.5-fold) (Table 5). For the Ni_1 and Ni_2 levels, treatments including B, MB, MB+M, and MB+B significantly enhanced the Ni content in the Res fraction relative to the control. Specifically, under Ni_1 conditions, the combined MB+M treatment exhibited the highest Ni accumulation (+36.6%). In contrast, at the Ni_2 level, MB alone produced the greatest increase (+29.6%), though their effects did not differ significantly from that of the MB+B treatment (Table 5). The results suggest that as soil Ni contamination levels increase, the effectiveness of soil amendments in promoting Ni retention in the Res fraction appears to diminish (Table 5). Pearson correlation analysis revealed a significant positive relationship ($r=0.42$, $P<0.01$) between Ni concentration in the Res fraction and soil Olsen-P content. This correlation suggests that elevated phosphorus levels in the soil solution, resulting particularly from MB and M amendments (which contain high P concentrations as shown in Table 3), may enhance Ni presence in the Res fraction through the formation of insoluble Ni-phosphate compounds (Boostani et al., 2023). Biochar produced from biomass, particularly livestock manure and municipal solid waste enhance the bioavailability of P in soil upon application (Shi et al., 2023).

Finally, the sequential fractionation analysis of soil Ni revealed that all amendments except M promoted Ni immobilization by converting more accessible fractions (WsEx, Car, and FeMnOx) into the Res form. Notably, combined application of amendments including MB+M, MB+B ad M+B demonstrated no superior immobilization efficacy compared to individual treatments, with MB (biochar) emerging as the most effective amendment for Ni stabilization. Conversely, the M

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(compost) treatment appeared to enhance Ni mobility by increasing its concentration in bioavailable fractions (WsEx and Car). Mailakeba and Bk (2021) investigated the effects of incorporating kunai grass biochar at a rate of 0.75% into soils with varying Ni contamination levels (0, 56, 100, and 180 mg Ni kg^{-1} soil). Their findings indicated that the biochar amendment enhanced Ni retention in the Res fraction while decreasing its presence in other soil fractions. Similarly, Boostani et al. (2023) reported that the addition of three different biochars derived from cow manure, municipal solid waste compost, and licorice root pulp, each applied at 3% (w/w), to a Ni-contaminated soil resulted in elevated Ni concentrations in the OM and Res fractions, alongside a reduction in Ni levels within the WsEx, Car, and FeMn oxide fractions.

3.6 Desorption pattern of soil Ni extracted by DTPA over time

The influence of various amendments on Ni release by DTPA solution over a 24-hour period is illustrated in Figure 5. Across all treatments, Ni desorption exhibited an initially rapid rate within the first two hours, followed by a slower release phase before reaching equilibrium by 24 hours (Figure 5). This biphasic pattern suggests that Ni desorption from the soil occurs in two distinct stages, each associated with different bonding energies (Boostani et al., 2019). The initial phase likely corresponds to more readily available Ni fractions, such as WsEx and Car forms, whereas the subsequent phase may be attributed to Ni associated with less labile fractions, including FeMnOx and OM pools (Boostani et al., 2023). Similar biphasic HMs release patterns have been frequently documented in previous studies (Jalali et al., 2019; Sajadi Tabar and Jalali, 2013; Taghdis et al., 2016).

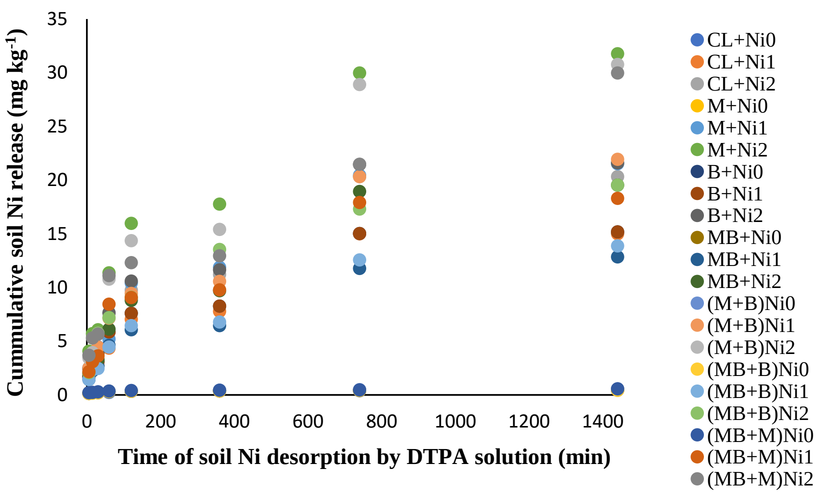

Figure 5. Cumulative soil Ni release (mg kg^{-1}) by DTPA solution over time.
Notes: Cl, control (without amendment addition); M, municipal solid waste compost; B, bentonite clay; MB, municipal solid waste compost biochar; Ni_0, without Ni application; Ni_1, application of 150 mg Ni kg^{-1} soil; Ni_2, application of 300 mg Ni kg^{-1} soil

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Elevating Ni concentrations from Ni_0 to Ni_2 resulted in a substantial increase in Ni release. Additionally, all amendments except M contributed to a reduction in soil Ni desorption (Figure 5). The highest cumulative Ni release over 24 hours was observed in the M×Ni_2 treatment, showing a 56.2% increase compared to the Cl×Ni_2 treatment. Conversely, the lowest Ni desorption at both Ni_1 and Ni_2 levels was recorded in the MB treatment, with values of 12.84 mg Ni kg^{-1} and 19.52 mg Ni kg^{-1}, respectively. These findings reaffirm that MB was the most effective treatment for Ni immobilization, whereas the sole application of M enhanced Ni mobility. However, the combined application of M with B or MB moderated and reduced Ni release compared to M alone at all the Ni levels (Figure 5). In a study, the minimal Cd release from a contaminated calcareous soil over a 24-hour period was recorded in the co-application treatment of municipal solid waste biochar and bentonite. This represented an 18.7% decrease in Cd desorption relative to the control (Boostani et al., 2025).

3.7 Application of power function kinetics models to soil Ni desorption data

The effectiveness of various amendments in immobilizing Ni in soil was also evaluated using power function kinetic model. The release of Ni from soil, when extracted with DTPA solution over time, conformed to the power function kinetics model, demonstrating strong model fit with determination coefficients (R^2) ranging from 0.86 to 0.99 and standard errors of estimate (SEE) between 0.08 and 0.18. These findings align with prior research establishing the power function equation as the most appropriate kinetic model for characterizing the desorption behavior of HMs in calcareous soils (Zahedifar and Moosavi, 2017; Sheikh-Abdullah et al., 2021; Boostani et al., 2025). The power function equation can be mathematically represented in two forms: $q_t = at^b$ or in its linearized logarithmic form: $Ln\, q_t = Ln\, a + b\, Ln\, t$, where ‘$q_t$’ shows cumulative Ni desorbed at time t (mg Ni kg^{-1} soil), ‘a’ indicates initial Ni desorption rate (mg Ni kg^{-1} min^{-1}), and ‘b’ represents desorption rate coefficient (mg Ni kg^{-1})$^{-1}$. The interaction effects of soil amendments and Ni rates on changing the magnitude of parameters derived from power function kinetic model were statistically significant ($P < 0.01$). The kinetic parameters (‘a’ and ‘b’) exhibit an inverse relationship with desorption behavior, where decreasing ‘a’ values and increasing ‘b’ values correspond to suppressed HMs release rates, as demonstrated by Dang et al. (1994). Among the single amendment treatments, MB consistently demonstrated the lowest ‘a’ parameter values and highest ‘b’ parameter values across all Ni concentration levels (Table 6), indicating its superior efficacy in reducing Ni desorption from soil. Regarding combined amendments, the MB+B treatment showed optimal performance in suppressing Ni release based on kinetic parameters, though statistical analysis revealed no significant difference between MB+B and MB alone (Table 6). The power function equation can be differentiated with respect to time (t) to obtain the instantaneous desorption rate: $\frac{d_q}{d_t} = a.b.t^{b-1}$, at t = 1, this expression reduces to: $\frac{d_q}{d_t} = ab$, where the product ‘ab’ corresponds to the initial desorption rate of the element from the soil matrix (Dalal, 1985). At the Ni_0 level, none of the applied amendments exhibited a statistically significant influence on the ‘ab’ parameter (Table 6). However, at the Ni_1 level, treatments containing M (M, M+B, and M+MB) demonstrated a significant enhancement in ‘ab’ parameter values (Table 6). In contrast, under Ni_2 conditions, individual applications of B and MB, as well as their combination (MB+B), significantly reduced the ‘ab’ parameter values relative to the control. The most

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substantial reduction (30.3%) was observed for the MB treatment (Table 6). A statistically significant and strongly positive correlation was observed between the 'ab' parameter and both the Ni-WsEx (r=0.90, P<0.01) and Ni-Car (r=0.97, P<0.01) fractions. These findings indicate that the initial release kinetics of Ni are governed by its concentration in the more labile fractions characterized by weaker adsorption energies.

Table 6. The Parameters of power function kinetics model as affected by the application of different amendments

	Cl	M	B	MB	M+B	MB+B	MB+M	
				a (mg Ni kg^{-1} min^{-1}) ^{b}				
Ni_0	0.12^{h}	0.12^{h}	0.11^{h}	0.091^{h}	0.11^{h}	0.095^{h}	0.15^{h}	0.11^{C}
Ni_1	0.83^{g}	1.22^{c-e}	0.90^{f-g}	0.74^{g}	1.27^{cd}	0.78^{g}	1.15^{d-f}	0.98^{B}
Ni_2	1.47^{bc}	2.05^{a}	1.23^{c-e}	0.96^{e-g}	1.57^{b}	1.21^{c-e}	2.02^{a}	1.50^{A}
	0.80^{C}	1.13^{A}	0.74^{C}	0.61^{D}	0.98^{B}	0.68^{CD}	1.10^{AB}	
				b (mg Ni kg^{-1})$^{-1}$				
Ni_0	0.18^{g}	0.19^{fg}	0.21^{ef}	0.23^{e}	0.23^{e}	0.23^{e}	0.18^{g}	0.21^{B}
Ni_1	0.41^{a-c}	0.40^{a-c}	0.40^{a-c}	0.39^{c}	0.39^{c}	0.41^{a-c}	0.40^{a-c}	0.40^{A}
Ni_2	0.40^{a-c}	0.38^{c}	0.41^{a-c}	0.42^{a}	0.42^{a}	0.40^{a-c}	0.36^{d}	0.40^{A}
	0.33^{B}	0.33^{B}	0.34^{A}	0.35^{A}	0.35^{A}	0.35^{A}	0.32^{B}	
				ab				
Ni_0	0.021^{h}	0.025^{h}	0.022^{h}	0.021^{h}	0.025^{h}	0.021^{h}	0.027^{h}	0.023^{C}
Ni_1	0.338^{g}	0.491^{de}	0.365^{fg}	0.307^{g}	0.506^{de}	0.302^{g}	0.457^{ef}	0.395^{B}
Ni_2	0.587^{cd}	0.797^{a}	0.503^{de}	0.409^{e-g}	0.663^{bc}	0.488^{de}	0.723^{ab}	0.595^{A}
	0.315^{B}	0.438^{A}	0.297^{BC}	0.245^{C}	0.398^{A}	0.270^{BC}	0.402^{A}	

Notes: Cl, control (without amendment addition); M, municipal solid waste compost; B, bentonite clay; MB, municipal solid waste compost biochar; Ni_0, without Ni application; Ni_1, application of 150 mg Ni kg^{-1} soil; Ni_2, application of 300 mg Ni kg^{-1} soil. Numbers with same lower letters (interaction effects), in each section, had no significance difference to each other (P < 0.05). Also, numbers with same capital letters (main effects), in each section, had no significance difference to each other (P < 0.05).

4 Conclusions

This study evaluated the effectiveness of various organic and inorganic amendments including biochar (MB), compost (M), and bentonite (B) applied both individually and in combination, for Ni immobilization in a calcareous soil artificially contaminated with different Ni concentrations. Sequential extraction confirmed that all amendments except M promoted Ni immobilization by converting labile fractions (WsEx, Car, FeMnOx) into the Res form. Notably, combined treatments (MB+M, MB+B, M+B) did not exhibit synergistic effects, with MB alone emerging as the most effective amendment. Desorption kinetics analysis supported these findings, with MB demonstrating the lowest cumulative Ni release and the highest retention capacity, as evidenced by its favorable power function kinetics model parameters (low 'a' and high 'b' values). This outcome was probably attributed to MB's alkaline properties, high ash content, and phosphorus levels, which facilitated Ni precipitation as hydroxides, carbonates, and phosphates. In contrast, M increased Ni mobility by enhancing its presence in the bioavailable fractions of WsEx and Car. Finally, this study demonstrates that municipal solid waste biochar (MB) is a highly effective amendment for Ni immobilization in contaminated calcareous soils, outperforming both individual and combined applications of compost and bentonite. These findings provide valuable insights for developing remediation strategies in Ni-contaminated agricultural soils, emphasizing the potential of biochar as a sustainable and efficient immobilization agent. Further research should explore long-term field applications to validate these findings under plant cultivation.

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