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Letter

Enhanced Rock Weathering Affects Formation of Mineral-Associated Organic Carbon in Soil

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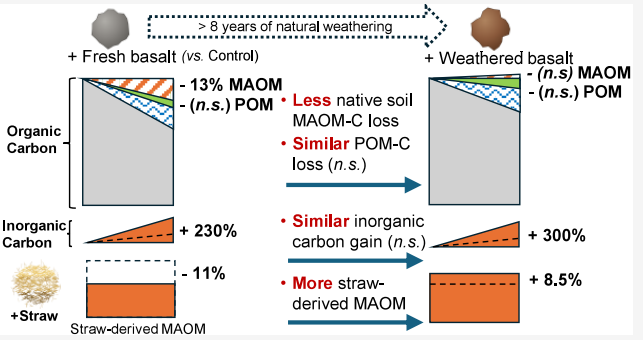
ABSTRACT: Enhanced silicate rock weathering (ERW) is increasingly considered for climate mitigation through inorganic carbon (IC) formation, yet its effects on soil organic carbon (OC) turnover remain poorly constrained and mechanistically unresolved. We investigated how the basalt weathering state influences mineral-associated organic matter (MAOM) in a 6 month microcosm incubation of a slightly acidic Cambisol. Fresh basalt (olivine-rich) and naturally weathered basalt (olivine-depleted and phyllosilicate-enriched) were applied, and ^{13}C-labeled straw was used to trace the incorporation of plant-derived C into MAOM. Fresh basalt caused more native MAOM-C loss (~13%) by 0.86 ± 0.29 mg of C (g of soil)$^{-1}$, likely driven by pH, whereas weathered basalt caused minimal pH change and marginal native MAOM-C loss (~0%) compared to the control. In ^{13}C-labeled straw-amended soils, weathered basalt, characterized by a larger specific surface area and limited pH alteration, increased straw-derived new MAOM-C formation by 20% and 8.5% compared to fresh basalt and the control, respectively. These microcosm incubation results suggest that basalt weathering within 6 months of application may strongly modulate native MAOM-C loss and new MAOM-C formation. Our findings indicate that ERW can influence soil MAOM-C beyond IC sequestration, which implies the need to include MAOM-C responses in ERW carbon accounting to improve its climate mitigation potential assessments.



KEYWORDS: *enhanced rock weathering, weathered basalt, organic carbon, mineral-associated organic matter, carbon sink*

1. INTRODUCTION

Enhanced rock weathering (ERW), typically involving silicate minerals, has primarily focused on inorganic carbon (IC) sequestration in terrestrial and aquatic systems.[1] These IC pools have been extensively modeled and quantified associated with ERW.[1−3] Recent findings suggest that ERW-associated processes may also affect organic carbon (OC) sequestration through alteration of soil pH, microbial processes, and organic matter (OM) decomposition rates,[4−12] which potentially affects the stabilization and formation of mineral-associated organic matter (MAOM),[5,9,11] an OC pool with longer turnover.[13,14] However, most ERW studies neglect this potential long-term MAOM accumulation, which means an overall incomplete evaluation of the C sequestration potential associated with ERW.

Weathering of ERW materials not only releases cations that allow the formation of IC by charge balance with bicarbonate but also may subsequently form secondary clay minerals with other weathering products.[8,15] This potentially means additional specific mineral surface area (SSA) in the soil, which may add to its potential OC stabilization potential.[4] This mechanism aligns with theoretical frameworks suggesting that soil carbon storage capacity depends on mineral content and composition.[16,17] These additional reactive mineral surfaces offer additional sorption sites for organo-mineral associations, promoting the carbon sequestration capacity over time.[18] However, the ERW-induced soil pH changes can also trigger pH-dependent shifts in organo−mineral associations, which are generally thought to be more stable under acidic conditions due to the formation of inner-sphere complexes and less stable at near-neutral and neutral pH. With the increasing pH in different soils, divergent impacts on MAOM pools were observed in available ERW studies.[5−7,9,11,19] Besides the pH effect, the increased level of exchangeable cations from silicate weathering may sustain MAOM through cation bridging.[4,5,8,11] Due to our limited mechanistic understanding of ERW-associated MAOM formation, the relative importance of ERW-

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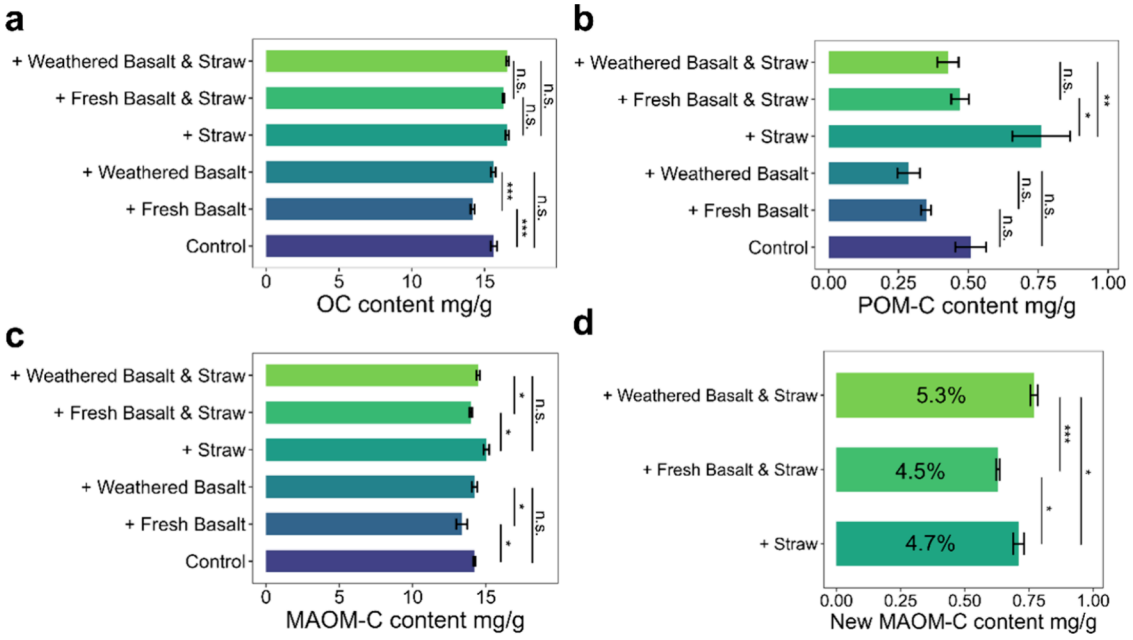

Figure 1. After incubation for 6 months, (a) organic carbon (OC) content derived from ref 4, (b) particulate organic matter carbon (POM-C) content, (c) mineral-associated organic matter carbon (MAOM-C) content, and (d) straw-derived new MAOM-C content in soils. The percentage means the relative contribution of straw-derived new MAOM-C in total MAOM-C content. Data are means ± SE (n = 4). Based on Tukey HSD post hoc analysis, *0.01 < p < 0.05, **0.001 < p < 0.01, and ***p < 0.001. The nonsignificant comparison is marked as n.s. Detailed data and statistical information can be found in Tables S3 and S4.

associated pH and mineral capacity in controlling MAOM dynamics remains unresolved.

To address this knowledge gap, we studied the effects of basalt application to a slightly acidic temperate Cambisol in Germany. To simulate long-term weathering effects, we applied basalts at two different weathering stages (fresh vs weathered). We also employed ^{13}C-labeled straw and density fractionation to distinguish native MAOM-C from newly formed MAOM-C, enabling the direct quantification of carbon transformation. This study aims to evaluate (a) whether the addition of fresh basalt alters native MAOM-C pools in slightly acidic soils and (b) how weathered basalt, distinguished from fresh basalt by differences in pH, specific surface area (SSA), exchangeable cations, and mineral composition, influences the formation of newly stabilized MAOM-C. Taken together, these results provide insight into whether progressive ERW may increase the soil capacity for long-term OC stabilization through multiple, potentially interacting processes.

2. MATERIALS AND METHODS

2.1. Microcosm Experiment Setup and Material Properties

A 6 month microcosm (5 cm height, ø 8 cm) experiment was conducted at 25 °C in darkness using slightly acidic temperate cropland topsoils (Cambisol) taken in southeastern Germany (49°06′12″N, 12°21′37″E). The soil parent material is Regensburg granite. The soil pH (0.1 M $CaCl_2$), total C content, OC content, SSA, silt content, and clay content were 5.8, 17 mg g^{-1}, 16.8 mg g^{-1}, 8.1 m^2 g^{-1}, 51%, and 18%, respectively as described by Lei et al.[4] Distinct from that work, this study focuses on the ERW effect of native and new OC formation and stabilization at different weathering stages.

Defined fresh basalt (olivine-enriched; pH 7.0; inorganic C, 4.24 mg g^{-1}; total C, 4.4 mg g^{-1}; OC, 0.16 mg g^{-1}) and weathered basalt (olivine-depleted, phyllosilicate-enriched; pH 6.7; inorganic C, 0.05 mg g^{-1}; total C, 0.69 mg g^{-1}; OC, 0.64 mg g^{-1}) were sourced from Zeilberg quarry, previously classified as a nepheline basanite.[20] According to the total alkali-silica diagram,[21] the fresh basalt was classified as basanite. The two materials share the same quarry, lithology, and immobile-element signatures (Ti), consistent with a common parentage, though we cannot fully exclude compositional heterogeneity between volcanic flows or sill levels. The weathered basalt represents a more advanced weathering stage of the same lithology, as evidenced by an increase in the chemical index of alteration (CIA), loss of primary nepheline reflections, and enrichment of poorly crystalline clay minerals (Table S1 and Figure S1).

Both materials were milled to a fine particle size and applied at 4% (w/w) to simulate long-term weathering of basalt responses in natural soils (Table S1). The milling process primarily aims to disaggregate sandy-sized to silty-sized fractions, and there is a marginal (<1%) difference in the clay size fraction after milling. Additional ^{13}C-labeled straw (δ^{13}C, 238‰ V-PDB) was added at a weight ratio of 0.8% to trace new plant-derived OM. The dry straw was ball-milled under liquid nitrogen cooling to fine sizes (1.5 min at 25 Hz). Inorganic C in soils was removed before isotopic analysis.

Soil moisture was maintained above 50% throughout the incubation period. To maintain the moisture content, in addition to the weekly 84 mL of distilled water, 20−30 mL of irrigation was applied in between.

After incubation for 6 months, bulk soils (<2 mm) were sampled and dried in an oven at 40 °C. The bulk soil physicochemical properties (pH, total C, OC, SSA, exchangeable cations, and particle size distribution) can be found in ref 4 and Table S4.

2.2. Density Fractionation

Bulk soils (<2 mm) were density fractionated using sodium polytungstate (SPT, 1.6 g/cm^3) and sonication (400 J/g) to disperse soils, break aggregates, and release occluded particulate organic matter (POM). Soil fractionation was performed on four replicates due to the low variance in OC among the five original replicates and the time-intensive nature of the procedure. POM (<1.6 g/cm^3, free and occluded POM) and MAOM (>1.6 g/cm^3) fractions were pressure-filtered (0.45 μm) to remove SPT and recover solid fractions. The residues were freeze-dried and analyzed for total C, OC, IC, and δ^{13}C. No significant difference was found in mass or OC recovery between amendments. Straw-derived new MAOM-C was quantified via ^{13}C

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isotopic mixing models, enabling separation of native MAOM-C mobilization from new MAOM-C formation. No additional particle size fractionation was used. Prior work has shown that MAOM fractionated by density fractionation exhibits the same turnover rates as the MAOM isolated by particle size fractionation, confirming they capture the same slow-cycling OC pool.[22] Soil mass recovery exceeded 97%, and OC recovery was above 90% (Table S2).

2.3. ^{13}C Isotopic Mixing Model and Calculations

The proportion of straw-derived MAOM-C formed (f) in the soil was calculated using the equation

$$f = \frac{\delta_t - \delta_s}{\delta_r - \delta_s}$$

where δ_t is the $\delta^{13}C$ of the MAOM-OC fraction with additional basalts and straw after carbonate removal, δ_s is the $\delta^{13}C$ of the MAOM-OC fraction after carbonate removal in their respective paired basalt amendments without straw, and δ_r is the $\delta^{13}C$ of the added straw. All samples were measured in triplicate by isotope ratio mass spectrometry (IRMSdelta V Advantage, Thermo Fisher, Dreieich, Germany). The addition of 4% rock amendment (w/w) dilutes the OC pools. We corrected for this by calculating the MAOM-C content relative to the total amended soil dry mass (soil + basalt amendments). Specifically, the amended soil was assumed to comprise 96% OC-only containing soil (no significant difference between total C and OC in control soils) and 4% basalt materials. The MAOM-C contents were expressed relative to the soil-only mass basis (measured value × 0.96). All reported MAOM-C content reflects this composition.

2.4. Statistical Analysis

The statistical analysis was conducted in R (version 4.3.1) (R Core Team, 2013), using packages including 'readxl', 'ggplot2', 'paletteer', 'dplyr', 'tidyverse', 'car', and 'multcomp'.

To compare the significance of the OC content, POM-C content, MAOM-C content, and new MAOM-C content between amendments, a two-way ANOVA was conducted to perform pairwise comparisons at a significance level of 0.05, followed by a Tukey HSD test for post hoc analyses. The Levene test and Shapiro–Wilk test were performed to validate data set homogeneity and assess the normality of residuals in the model, respectively. Simple and multiple linear regression was used to test relationships among pH, SSA, and exchangeable Ca^{2+} and MAOM-C (native MAOM-C and straw-derived new MAOM-C). To assess whether the effect of pH depended on exchangeable Ca^{2+}, an interaction effect (pH × exchangeable Ca^{2+}) was included. Predictors were mean-centered prior to forming the interaction term to reduce multicollinearity and variance inflation.

3. RESULTS AND DISCUSSION

3.1. Fresh Basalt Destabilizes Native MAOM

Fresh basalt amendment caused a significant decrease in native MAOM-C content compared to the control soil after incubation for 6 months (Figure 1). Native MAOM-C decreased by 0.86 ± 0.29 mg of C (g of soil)$^{-1}$, which represents an approximately 13% loss of the overall MAOM-C pool. This decrease directly correlated with fresh basalt triggering an increase in the soil pH from ~5.8 to ~6.1 due to the presence of rapidly dissolving calcite and olivine (Figure 2a) and may also be influenced by the exchangeable Ca^{2+} content in the soil (Figure S1). No difference was observed between soils amended with weathered basalt and the control soils (Figure 1c).

The decrease in MAOM-C content was consistent with the observed bulk OC decrease as reported by Lei et al.,[4] who found soil pH was one of the primary factors leading to bulk OC losses. This study further supports that pH-induced OC

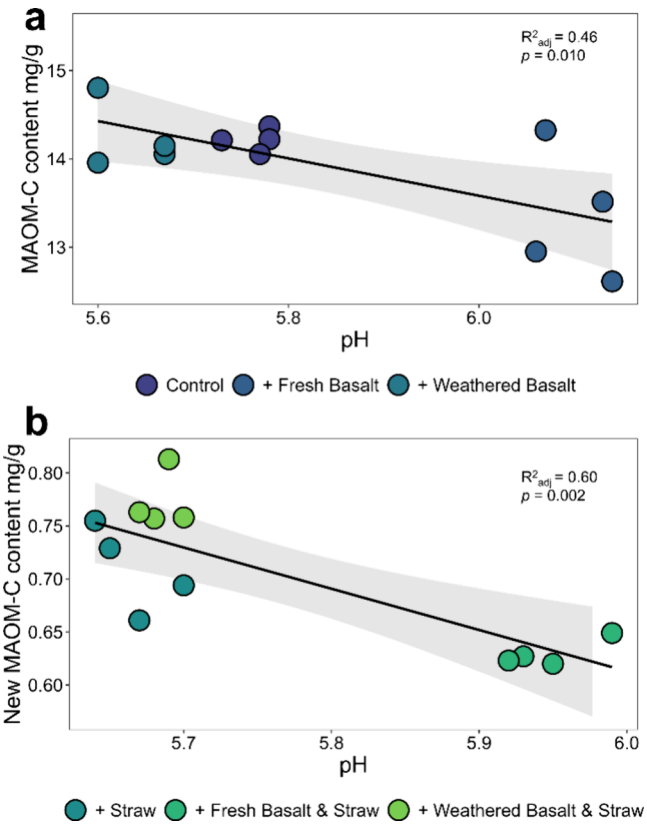

Figure 2. Single-linear regression models for (a) native MAOM-C and pH and (b) new MAOM-C and pH in straw-amended soils. Regression fits shown with 95% confidence intervals. The pH, specific surface area, and exchangeable Ca^{2+} data were sourced from ref 4. The two single linear models explain 46% and 60% of the variance in native and new MAOM-C, respectively. Other detailed statistical information can be found in Table S5.

losses by fresh basalt may be primarily through losses of slower cycling MAOM-C pools, while SSA showed no correlation with native MAOM-C (Table S5), although the increased SSAs are suspected to be positively related to the MAOM pool. The pH-dependent MAOM loss with fresh basalt may reflect a shift in organo–mineral associations, potentially from inner-sphere complexes (dominant at pH 5.8) to outer-sphere complexes (favored at pH 6.1) that reduce bonding strength,[13,20] and a pH-induced microbial decomposition acceleration as soil pH enters a range more favorable for soil microbial communities.[24] This may explain why the loss of OC in bulk soils is primarily reflected in the MAOM fraction. In contrast, weathered basalt showed only marginal pH change (less than 1 unit) and provided increased exchangeable Ca^{2+}, potentially facilitating cation bridging. Although the released Fe^{3+} and Al^{3+} may also facilitate organo–mineral interactions, we expect their effect to be minor considering their quantity and much slower dissolution rate (Table S1). In addition, the correlation between the interaction effect (pH × exchangeable Ca^{2+}) and native MAOM-C was significant ($p < 0.05$) (Table S5 and Figure S1), indicating that the relationship between pH and native MAOM-C may be dependent on exchangeable Ca^{2+}, which explain 67% of the variance (Table S5). This shows a negative correlation between native MAOM-C and pH in soils with low exchangeable Ca^{2+}. However, the negative relationship between native MAOM and pH was weakened or even became positive at a higher Ca^{2+} level (Figure S1). This

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may suggest that a greater supply of Ca^{2+} could partially offset pH-associated MAOM losses. Given our sample size (n = 12), this interaction should be considered exploratory and warrants confirmation in larger data sets.

A lower POM-C content in fresh and weathered basalt-amended soils was also observed compared to the control soils (Figure 1b), though it was not significant (p > 0.05 (Table S3)). This can still reflect a trend that the mineralization of the POM fraction is accelerated compared to the control soils in both basalt amendments, which may be due to the optimized pH and additional nutrient supply (e.g., P, K, Ca, and Mg).

Therefore, this study suggests that the fresh basalt amendment can lead to a bulk OC loss over the short term (6 months) primarily in the form of the native MAOM-C pool in slightly acidic soils potentially due to significant acid neutralization, which may shift the surface protonation and accelerate microbial mineralization. Exchangeable Ca^{2+} may modulate this response (significant pH × Ca interaction (Table S5 and Figure S2)), but its main effect was weak (Table S5). In contrast, the weathered basalt can maintain native MAOM-C likely due to the marginal soil pH shift and an additional supply of exchangeable Ca^{2+}.

3.2. Weathered Basalt Enhances Plant-Derived New MAOM Formation

In straw-added amendments, this study showed significantly enhanced retention of straw-derived new OC in weathered basalt amendments (5.3%; 0.77 ± 0.01 mg of C (g of soil)$^{-1}$) and the straw-only soils (4.7%; 0.71 ± 0.02 mg of C (g of soil)$^{-1}$) compared to the fresh basalt amendments (4.5%; 0.63 ± 0.01 mg of C (g of soil)$^{-1}$). Specifically, the weathered basalt retaining newly added straw-derived MAOM-C surpassed that of unamended soils by 8.5 ± 3.7% and fresh basalt amendments by 20.0 ± 2.2% (Table S4). Straw-derived new MAOM-C was negatively associated with pH (p < 0.05 (Figure 2b)) and showed a positive association with SSA (p < 0.1 (Table S5)), whereas relationships with exchangeable Ca^{2+} were weak (Table S5).

This observed enhanced new OC retention in MAOM pools in weathered basalt amendments may reflect a combined effect of the minimal pH change and additional highly reactive mineral surfaces, shown as SSA (Table S5), which coexplained 76% of the variance of the new MAOM-C changes. A larger SSA provided by additional clay minerals (12% of clay in weathered basalt), including smectites, vermiculites, and Fe/Al oxides (Figure S1),[4] can provide more sorption sites compared to fresh basalt (~1% clay) and control soils.[18] For fresh basalt, the pH shift could weaken organo−mineral bonds and stimulate microbial activity; both could hamper the accumulation of MAOM on mineral surfaces. While fresh basalt may destabilize organo−mineral complexes and promote microbial mineralization, promoted necromass production could partially compensate by providing alternative stabilization. Though this additional microbial biomass/necromass may partially offset this effect, the extent remains unquantified. The combined effect of a stable pH and a larger SSA in weathered basalt suggest more favorable conditions for new MAOM retention, while the role of additional exchangeable Ca^{2+} is negligible (Table S5). However, we cannot distinguish whether the straw-derived new OC is associated with newly exposed mineral surfaces due to basalt weathering or may have interacted with the native preexisting mineral surface area, which requires further mineralogical and spectrometry analysis.

These factors are critical during the transformation from plant residues to the MAOM-C pools. Besides the pH effect, considering the fact that a larger SSA is correlated with greater MAOM stabilization[25] and the silicate minerals can form highly reactive secondary phyllosilicates and amorphous metal oxides, including smectites, vermiculites, and Fe/Al oxides,[26,27] these guarantee that the weathering of silicate minerals shows a strong potential necessary for subsequent MAOM pool accrual.[16]

Thus, we observe the fact that the observed lower retention of new MAOM-C derived from plant residues in fresh basalt-amended soils and the enhanced new MAOM-C formation in weathered basalt-amended soils are likely attributed to distinct changes in soil pH and SSA. We posit that the enhanced accumulation of new MAOM-C in weathered basalt may represent a continuous buildup potential of slowly cycled C pools with fresh basalt weathering, which indicates a long-term C pool alongside the well-documented IC accrual associated with the ERW strategy.

3.3. Implications for ERW Carbon Accounting with Weathering Progressing

Our results demonstrate that the stage of basalt weathering fundamentally alters its short-term impact on the MAOM-C pools. Fresh basalt induced a significant loss of native MAOM-C, which was correlated to an increase in pH. Weathered basalt enhanced the retention of plant-derived new MAOM-C formation, correlating with its larger SSA and marginally changed soil pH. This contrast suggests that the net effect of ERW on the MAOM-C pool may be dependent on the weathering stage. An initial phase following fresh rock application could be characterized by a net loss of native MAOM due to rapid acid neutralization, particularly in slightly acidic soils, whose mineral surfaces can be more easily deprotonated with an increase in pH. The concurrent IC gain in soil and leachate observed by Lei et al.[4] cannot compensate for this part of MAOM-C loss, resulting in net C loss from the system. In contrast, in weathered basalt, the formation of secondary, large-SSA minerals (phyllosilicates and oxides) creates additional highly reactive mineral surfaces conducive to the accumulation of new MAOM, which results in both IC gain[4] and new MAOM-C formation in soils. The weathered basalt, enriched in secondary minerals and preassociated OC (Table S1), serves as a proxy for this advanced weathering stage. This indicates that similar effects may also occur in fresh basalt amendments after weathering for several years. With continuous inputs of OM in natural soils from aboveground and belowground biomass, organic amendments, and microbial residues, the potential for OC stabilization as MAOM-C may be significantly enhanced with the weathering of fresh ERW materials (Figure 1d). The limited alkalizing potential of weathered ERW materials on soils, compared to those of fresh ones potentially causing temporary OC losses in slightly acidic soil,[4,23] can ultimately stabilize OM associated with minerals. However, this may not be the case in alkaline soils, where the ERW-induced pH effect is expected to be less pronounced due to high base saturation and the potential existence of carbonates.[9] The pH-related shift in organo−mineral interactions and in microbial activity and carbon use efficiency may be expressed more on Ca-mediated cation bridging and aggregation, while the formation of secondary minerals may be slower due to the slower dissolution rate.

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Therefore, these findings suggest that the climate benefits of ERW may extend beyond the IC sink alone. A more comprehensive carbon accounting framework should therefore consider the potential accrual or loss of MAOM-C that may develop concurrently with mineral weathering. However, because the weathered basalt used in this study represents only a proxy for natural weathering, it does not capture the dynamic processes associated with the progressive weathering of fresh basalt in soils, nor does it demonstrate that continued weathering will necessarily offset the initial losses of native MAOM-C. Given the modest sample size (n = 12), though the interaction effects should be interpreted cautiously and warrant confirmation in larger data sets, we posit that the magnitude and persistence of MAOM-C accumulation are likely governed by complex interactions among soil acidity, texture, SSA, mineral composition, and the rates of secondary mineral formation. In addition, the high heterogeneity under field conditions may lead to some spatial hot spots for mineral dissolution, which cannot be captured in this microcosm study. Consequently, long-term field studies of ERW-amended soils, incorporating repeated pH measurements, detailed mineral characterization, particle size dynamics, and direct assessments of organo−mineral associations, are required to determine whether the short-term responses observed here can translate into sustained MAOM accumulation in natural ecosystems and to quantify its contribution to the overall carbon sequestration potential of ERW.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.estlett.6c00633.

Additional experimental details, materials, and methods: basalt classification and characterization, including XRF, CIA, and mineralogical information (Table S1) and XRD patterns (Figure S1); soil mass and OC recovery across amendments (Table S2); statistical tests for POM-C, MAOM-C, and new MAOM-C responses (Tables S3 and S4); and regression models for MAOM-C versus pH, SSA, and exchangeable Ca^{2+} (Table S5) (PDF)

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Notes

The authors declare no competing financial interest.

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