

## RESEARCH PAPER

# Does biochar contribute to close nutrient cycles of tree plantations on degraded Ultisols in the Ecuadorian Amazonia?

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## Abstract

The use of biochar is expected to improve soil fertility and close nutrient cycles in degraded strongly weathered tropical soils. We, therefore, hypothesized that biochar amendment to tree plantations (a) increases nutrient fluxes with litterfall alone and with mineral fertilizer plus lime and (b) reduces N losses reflected by lower  $\delta^{15}\text{N}$  values of litterfall and soils than in unamended controls. We grew the native leguminous *Schizolobium parahyba* var. *amazonicum* (Ducke) Barneby and the exotic *Gmelina arborea* Roxb at two sites. We used a replicated full factorial split-split plot design of amendment of mineral fertilizer plus lime, 3 and 6 t ha<sup>-1</sup> biochar, and a control. We collected litterfall biweekly (2012–2013) and topsoil samples (0–0.25 m) in 2009 before tree planting, in 2011 and 2013. Fertilizer plus lime increased the mean annual concentrations of P, Ca and Zn in litterfall but decreased that of Mn. At the same time, fertilizer plus lime increased the annual fluxes of nutrients, Na and Al with litterfall. During the dry season, biochar decreased the N concentration in litterfall and the K flux with litterfall. During the rainy season, biochar increased the concentrations of Ca and Zn in litterfall and their fluxes with litterfall. Biochar did not influence the  $\delta^{15}\text{N}$  values of soil and litterfall after 51 months of tree growth. Fertilizer plus lime decreased the  $\delta^{15}\text{N}$  values of soil, because of the lower  $\delta^{15}\text{N}$  value of the used urea (−0.30‰) than the soil (4.5‰–7.8‰). Moreover, fertilizer plus lime increased the  $\delta^{15}\text{N}$  values of litterfall, possibly because of enhanced <sup>14</sup>N leaching from the N-rich canopies. The amendment of up to 6 t ha<sup>-1</sup> biochar did not contribute to close nutrient cycles.

## KEYWORDS

liming, litterfall, mineral fertilization, split-split plot design, stable nitrogen isotope ratios

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## 1 | INTRODUCTION

Biochar is a product of biomass pyrolysis under limited oxygen supply and at high temperature (Lehmann, 2007; Lehmann et al., 2006). It has been widely used as an amendment to increase soil quality (Bai et al., 2015; Barrow, 2012; Chan et al., 2008; Ding et al., 2016; Song et al., 2016). Biochar can improve soil physical properties, increase soil pH and cation-exchange capacity (CEC) (Chan et al., 2008; Criscuoli et al., 2014; Hernandez-Soriano et al., 2016; Kookana et al., 2011) and serve as a slow-release fertilizer (Hu et al., 2019; Shi et al., 2020). Moreover, biochar can enhance nutrient retention (Deluca et al., 2015; El-Naggar et al., 2019; Novotny et al., 2015). It has been reported that biochar amendment to soils can increase the retention of N in forms that are bioavailable in the short and intermediate term and reduce N leaching (Drake et al., 2015; Nelissen et al., 2012; Pratiwi et al., 2016; Zhang et al., 2017). Therefore, it was suggested that biochar can particularly be used to remediate strongly weathered, degraded, nutrient-poor tropical soils including Oxisols and Ultisols (Glaser et al., 2000, 2001, 2002; Lehmann et al., 2003; Lima et al., 2002; Major et al., 2010).

An increased retention of bioavailable nutrients in soil following biochar amendment has been shown to be beneficial for plant growth (Arif et al., 2017; Haider et al., 2017; Mandal et al., 2016; Zhang et al., 2017) and should, therefore, facilitate the uptake and cycling of nutrients by plants. In forests, nutrient recycling from plant to soil occurs via nutrient leaching from the canopy (Levia & Frost, 2006; Velescu et al., 2021; Wilcke et al., 2001) and litterfall (Likens, 2013; Wilcke et al., 2002). The increased nutrient availability in soils after mineral fertilization can be reflected by increased litterfall, nutrient concentrations in litterfall and nutrient fluxes with litterfall (Homeier et al., 2012; Wullaert et al., 2010). Thus, the addition of biochar might similarly increase nutrient concentrations of litterfall and nutrient fluxes with litterfall (Lin et al., 2017).

An increased retention of N in the ecosystem after the amendment of biochar could be reflected by changing stable N isotope ratios because N losses by leaching or gaseous emissions shift the N isotope composition ( $\delta^{15}\text{N}$  values) of the soil N to higher contributions of the heavy  $^{15}\text{N}$  isotope (Dawes et al., 2017; Högberg, 2008; Kellman & Hillaire-Marcel, 2003; Pardo et al., 2006; Sebilo et al., 2006; Xia et al., 2020). This implies that the  $\delta^{15}\text{N}$  values are the lower, the more closed the N cycle is. In plant communities containing legumes, the  $\delta^{15}\text{N}$  values of soils and plants are also influenced by the  $\delta^{15}\text{N}$  value of  $\text{N}_2$  in the atmosphere, which is by convention 0‰. If the legumes fix  $\text{N}_2$  from the atmosphere via their symbiotic rhizobia, the  $\delta^{15}\text{N}$  value of soil and plants is shifted

towards 0‰ (Evans, 2001; Inglett et al., 2004). Because mineral fertilizers frequently show negative  $\delta^{15}\text{N}$  values, mineral fertilization can also decrease the  $\delta^{15}\text{N}$  values of soils and plants (Choi et al., 2017; Coplen et al., 2002; Park et al., 2015). As a consequence, in legume-containing and fertilized ecosystems  $\delta^{15}\text{N}$  values could decrease even if the N cycling does not become tighter. Because the  $\delta^{15}\text{N}$  values of litterfall reflect that of the soil (Ibell et al., 2013), they will change in the same direction as those of the soil (Reverchon et al., 2015; Vigulu et al., 2019). Besides resulting from a more open N cycle, higher  $\delta^{15}\text{N}$  values in litterfall might, however, also indicate that the plants take up N from greater soil depth, because the  $\delta^{15}\text{N}$  values usually increase with increasing depth (Högberg, 2008) instead of reflecting a more open N cycling.

In the same tree plantations as reported here, González Sarango et al. (2021) did not observe an effect of up to  $6 \text{ t ha}^{-1}$  biochar on the growth of the two tree species *Schizolobium parahyba* var. *amazonicum* (Ducke) Barneby and *Gmelina arborea* Roxb during 51 months on degraded Ultisols at two study sites in the Ecuadorian Amazonia. In contrast, the amendment of a complete fertilizer plus lime had a strong effect on tree growth. The same was true for the annual litterfall of both tree species measured for 1 year at a tree age of 3–4 years (González Sarango et al., 2021). At the inherently less fertile of the two study sites, biochar increased pH, effective cation-exchange capacity (ECEC) and base saturation (BS) significantly. Moreover, González Sarango et al. (2022) reported that biochar increased  $\text{NaHCO}_3$ -extractable Ca ( $p < 0.05$ ) and Zn ( $p < 0.1$ ) and total N concentrations ( $p < 0.05$ ) in the topsoil. Biochar increased Ca ( $p < 0.1$ ) and Zn ( $p < 0.05$ ) retention in mineral fertilized topsoils calculated as the difference between the initial element stock plus the amended nutrients with mineral fertilizer, lime and/or biochar and the final stock at the time of tree harvest. In unfertilized topsoils, the amendment of  $6 \text{ t ha}^{-1}$  of biochar decreased total N retention ( $p < 0.05$ ), which González Sarango et al. (2022) attributed to a slight change in bulk density because of the amendment of biochar. While the previous studies at the same study sites addressed biochar effects on plant productivity (González Sarango et al., 2021) and soil fertility (González Sarango et al., 2022), it remains unclear if biochar helps close nutrient cycling and thus improves the long-term efficiency of the initial soil preparation effort.

Therefore, our overall goal was to determine, whether the amendment of up to  $6 \text{ t ha}^{-1}$  biochar helped close nutrient cycling in plantations of two tree species at two differently fertile locations in the Ecuadorian Amazonia. We hypothesized that (a) the amendment of biochar alone and in combination with fertilizer plus lime will increase the nutrient and Na concentrations of litterfall and fluxes with litterfall relative to the control plots. Further, we

hypothesized that (b) the simultaneous amendment of biochar will reduce N losses from the mineral fertilized plots and therefore result in N-isotopically lighter litterfall and soils compared with the plots that received only fertilizer plus lime. In the latter plots, in contrast, the higher N loss will result in the accumulation of  $^{15}\text{N}$  in soil and litterfall.

## 2 | MATERIAL AND METHODS

### 2.1 | Study sites and selected tree species

The study sites were already described by González Sarango et al. (2021, 2022). The experiment was conducted at two sites (La Victoria and Los Zapotes) in the Province of Zamora-Chinchi in the south Ecuadorian Amazonia (Table S1). We selected the sites based on five criteria including (a) their location in the foothills and low-mountain ranges, where most degraded pasture soils occur in the Ecuadorian Amazonia, (b) typical parent rocks for the study area (i.e., granodiorite and andesite), (c) moderate-to-strong slope, which is characteristic for the degraded pastures of the study area, (d) elevation of 850–1000 m a.s.l. to fall into the native premontane forest zone, and (e) evidence of cattle ranching by the presence of the fern species *Pteridium aquilinum* (L.) Kuhn. The study soils were considered degraded because of the lack of an organic layer, the presence of only a thin A horizon in La Victoria, or even no A horizon in Los Zapotes, which indicated a strong erosion, because of the steep slopes. We planted the native leguminous *Schizolobium parahyba* var. *amazonicum* (Ducke) Barneby and the exotic *Gmelina arborea* Roxb at both sites. The tree species were selected because (a) they are fast-growing, (b) tolerate low-soil fertility, (c) and have a commercial value on the local market (Silva et al., 2011; Swamy et al., 2004).

### 2.2 | Experimental design

We used a replicated full factorial split-split plot design at each site following Dormann & Kühn (2011). The main plot was split into subplots for two tree species. The subplots were further split into with or without mineral fertilizer plus lime. The third and last split included three levels of biochar (0, 3, 6  $\text{t ha}^{-1}$ ), resulting in a total of 12 treatments. Each subplot had an area of 144  $\text{m}^2$  and was planted with 16 trees, which corresponds to 1111 trees  $\text{ha}^{-1}$ . At La Victoria, all main plots were replicated four times; at Los Zapotes, where the available experimental area was smaller than at La Victoria, all plots were replicated three times. We planted the trees into a circular

area with diameters of 2.4 and 2 m at La Victoria and Los Zapotes, respectively, after we had mixed the topsoil with the amendments to a depth of 0.25 m. The trees were planted from 3 to 7 August 2009 and harvested from 4 to 8 November 2013 after 51 months of growth.

As biochar amendment, we bought commercially available wood-derived charcoal on a local market in Loja, Ecuador. The wood was pyrolyzed in a traditional earthen kiln with small holes that could be opened and closed at temperatures of 200–500°C under low-oxygen concentration. This corresponds to the traditional local charcoal production practice. Before application, the biochar was milled to a coarse powder (<2 mm). The decision to apply 3 and 6  $\text{t ha}^{-1}$  of charcoal was based on the state of the literature at the time of the setup of the experiment (i.e., before 2009). We particularly relied on the only available other study, we were aware of, that used biochar in an afforestation (Kishimoto & Sugiura, 1985). Kishimoto & Sugiura (1985) had applied 0.5  $\text{t ha}^{-1}$  of charcoal annually during 5 years (i.e., 2.5  $\text{t ha}^{-1}$  in total). This charcoal amendment increased the height of *Cryptomeria japonica* D. Don trees by a factor of 1.26 to 1.35 and the biomass by a factor of 2.31–2.36.

Detailed site information, biochar properties and nutrient inputs with the used soil amendments are summarized in Tables S1–S3, taken from González Sarango et al. (2021, 2022).

### 2.3 | Measurements

Litterfall was collected biweekly from 1 March 2012 to 22 February 2013 with four 0.6 × 0.6 m litterfall traps per plot, dried in the oven at 60°C and weighed (González Sarango et al., 2021). Samples from the 0–0.25 m mineral soil depth layer were collected in March 2009 (before the start of the experiment), February 2011, and November 2013, when the trees were harvested. The soil samples were air-dried and sieved <2 mm. Aliquots of litterfall and soil samples were finely ground in a ball mill (MM 400; Retsch, Haan, Germany). All the samples were stored at a location with low humidity and temperature and without direct sunlight in closed polyethylene bags until analysis. Before processing them, we inspected them thoroughly for mould or the presence of insects but did not find any indication of sample degradation.

Total element concentrations in litterfall were determined after digestion with concentrated  $\text{HNO}_3$  in a microwave oven (MARS6Xpress, CEM, Kamp-Lintfort, Germany) by measurement with an inductively coupled plasma optical-emission spectrometer (ICP-OES, 5100 VDV; Agilent, Waldbronn, Germany). Accuracy was assessed by the analysis of the certified reference materials

**TABLE 1** Mean annual element concentrations and fluxes ( $\pm SD$ ) in litterfall collected between March 2012 to February 2013 for two tree species (*S. paraguayana* and *G. arborea*) and two study sites (La Victoria and Los Zapotes) irrespective of the treatment

Site	Tree species	Element concentrations												
		N	P	K	Ca	Mg	S	Fe	Mn	Zn	Cu	B	Na	Al
		mg kg <sup>-1</sup>												
La Victoria	<i>S. paraguayana</i>	17 ± 0.23 Aa	0.81 ± 0.03 Aa	3.0 ± 0.30 Bb	9.0 ± 0.75 Bb	1.6 ± 0.12 Bb	1.1 ± 0.80 Ba	0.18 ± 0.03 Ba	32 ± 4.0 Bb	20 ± 2.0 Ab	6.7 ± 0.51 Ba	57 ± 6.8 Aa	19 ± 1.9 Ba	0.36 ± 0.08 Ba
La Victoria	<i>G. arborea</i>	11 ± 0.12 Ab	0.90 ± 0.09 Aa	4.2 ± 0.38 Ba	12 ± 0.94 Ba	2.1 ± 0.18 Ba	0.80 ± 0.06 Bb	0.18 ± 0.02 Ba	110 ± 12 Ba	64 ± 5.4 Aa	5.5 ± 0.43 Ba	95 ± 11 Aa	13 ± 1.6 Bb	0.41 ± 0.06 Ba
Los Zapotes	<i>S. paraguayana</i>	15 ± 0.21 Aa	1.0 ± 0.06 Aa	2.7 ± 0.26 Ab	13 ± 0.66 Ab	2.6 ± 0.14 Ab	1.3 ± 0.06 Aa	1.0 ± 0.24 Aa	50 ± 3.9 Ab	18 ± 1.3 Ab	11 ± 0.67 Aa	63 ± 6.8 Aa	23 ± 2.6 Aa	1.3 ± 0.30 Aa
Los Zapotes	<i>G. arborea</i>	13 ± 0.19 Ab	1.2 ± 0.08 Aa	5.3 ± 0.37 Aa	15 ± 0.64 Aa	5.0 ± 0.23 Aa	1.1 ± 0.04 Ab	0.98 ± 0.20 Aa	200 ± 12 Aa	72 ± 4.0 Aa	7.9 ± 0.30 Aa	63 ± 7.5 Aa	19 ± 2.5 Ab	1.1 ± 0.20 Aa
Literature <sup>a</sup>	<i>S. paraguayana</i>	23	1.8	6.7	15	3.2								
Literature <sup>b</sup>	<i>G. arborea</i>	13	1.2	2.4	2.4	35	26	0.92	23	27	2.6			
		Element fluxes												
		g m <sup>-2</sup>												
La Victoria	<i>S. paraguayana</i>	0.23 ± 0.01 Bb	0.01 ± 0.001 Bb	0.04 ± 0.003 Bb	0.13 ± 0.01 Bb	0.02 ± 0.001 Bb	14 ± 0.9 Bb	2.4 ± 0.3 Bb	0.4 ± 0.03 Bb	0.28 ± 0.02 Bb	0.09 ± 0.01 Bb	0.7 ± 0.1 Ab	0.26 ± 0.02 Ba	4.5 ± 0.7 Bb
La Victoria	<i>G. arborea</i>	0.34 ± 0.02 Ba	0.029 ± 0.002 Ba	0.13 ± 0.01 Ba	0.38 ± 0.03 Ba	0.06 ± 0.004 Ba	26 ± 1.8 Ba	5.5 ± 0.5 Ba	3.7 ± 0.3 Ba	2.1 ± 0.2 Ba	0.17 ± 0.01 Ba	3.5 ± 0.3 Aa	0.34 ± 0.02 Ba	13 ± 1.4 Ba
Los Zapotes	<i>S. paraguayana</i>	0.31 ± 0.01	0.02 ± 0.001 Ab	0.05 ± 0.003 Ab	0.26 ± 0.01 Ab	0.05 ± 0.003 Ab	25 ± 1.3 Ab	18 ± 2.5 Ab	0.9 ± 0.06 Ab	0.3 ± 0.02 Ab	0.23 ± 0.02 Ab	1.1 ± 0.1 Ab	0.44 ± 0.03 Aa	22 ± 2.9 Ab
Los Zapotes	<i>G. arborea</i>	0.47 ± 0.02	0.04 ± 0.003 Aa	0.2 ± 0.01 Aa	0.58 ± 0.03 Aa	0.18 ± 0.01 Aa	43 ± 2.3 Aa	37 ± 4.2 Aa	6.9 ± 0.36 Aa	2.6 ± 0.14 Aa	0.29 ± 0.02 Aa	2.6 ± 0.2 Aa	0.69 ± 0.06 Aa	42 ± 4.7 Aa

Note: Moreover, element concentration ranges in litterfall from the literature for both tree species are shown. Different upper-case letters indicate significant differences between the study sites for the same tree species and different lower-case letters indicate significant differences between the tree species at the same site.

<sup>a</sup>Jaramillo Botero et al. (2008).

<sup>b</sup>Azeez (2019).

SRM1547 and SRM1515. Average recoveries  $\pm$  standard deviations were  $100\% \pm 10\%$  for all the elements. Precision determined by duplicate measurements was  $<5\%$ .

The  $\delta^{15}\text{N}$  values of ground aliquots of the litterfall and soil samples were measured with an Elemental Analyzer-Isotope Ratio Mass Spectrometer (EA-IRMS, Flash HT Plus-Delta V Advantage; Thermo Fisher Scientific, Waltham, MA, U.S.A.). The samples were calibrated with the help of the standards IAEA N1, IAEA N2 and USGS 25. To correct instrumental drift, we used acetaldehyde as an in-house standard. Every 10 samples were bracketed with acetaldehyde. The standard deviation of 12 measurements of acetaldehyde was  $<0.18\%$ .

## 2.4 | Statistical analyses

We evaluated our results with a Repeated Measures Analysis of Variance (ANOVA) for split-split plot designs using the software R. We used the packages *nortest* (Gross & Ligges, 2015) for tests of normality, *agricolae* (De Mendiburu, 2017) for ANOVA including the Tukey's HSD post-hoc test and *gplots* (Warnes et al., 2009) and *plotrix* (Lemon, 2006) for plotting data. Moreover, we ran additional ANOVAs on the mean element concentrations and  $\delta^{15}\text{N}$  values of litterfall and on the mean element fluxes of the rainy (March to September 2012, ca. 1800 mm of rainfall) and dry (October 2012–February 2013,  $<150$  mm of rainfall) seasons.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Nutrient cycling with litterfall

At tree ages of 3–4 years, when we determined litterfall, nutrient concentrations in litterfall and nutrient fluxes with litterfall, the annual litterfall of *S. parahyba* ranged from  $129 \pm 11$  to  $290 \pm 9.5 \text{ g m}^{-2}$  and that of *G. arborea* from  $291 \pm 19$  to  $499 \pm 16 \text{ g m}^{-2}$  (González Sarango et al., 2021). The element concentrations in our litterfall samples were comparable to those reported in the literature for *S. parahyba* (Table 1; Azeez, 2019; Jaramillo Botero et al., 2008). For *G. arborea*, similar N, P, K and Fe concentrations were reported but lower Ca, Mn, Cu and Zn and much higher Mg and S concentrations (Table 1; Azeez, 2019). We attribute the partly marked differences in the nutrient concentrations between our study and that of Azeez (2019) to different soil properties at their Nigerian savanna site than at our sites in the Ecuadorian tropical rain forest.

The site, site block and tree species had at least marginally ( $p < 0.1$ ) significant effects on all mean annual concentrations of nutrients, Na and Al in litterfall (Table S4). This reflected the higher inherent fertility and soil pH value

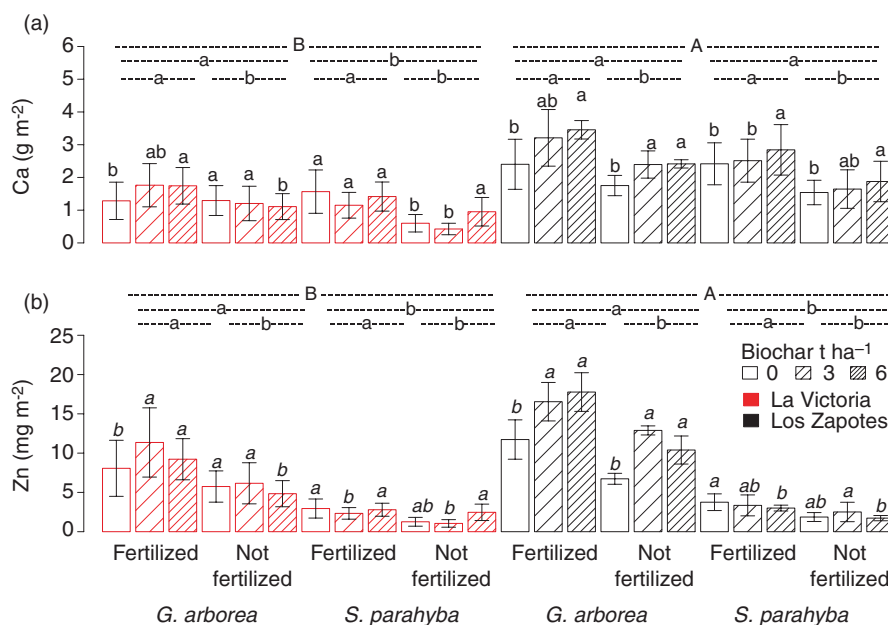
of the site at Los Zapotes than at La Victoria (González Sarango et al., 2021, 2022) and the higher fertility of the lower than the upper slope sites because of erosion. The genetically determined differences in nutrient concentrations between the two tree species might also have played a role. Fertilizer plus lime increased the mean annual concentrations of P, Ca and Zn in litterfall and decreased that of Mn (Table S4). Surprisingly, only the concentrations of three nutrients increased in spite of the amendment of a complete fertilizer (Table S3). This suggests that the P, Ca and Zn supply of the plants was particularly low in the studied degraded soils. We attribute the negative effect of fertilizer plus lime on mean annual Mn concentrations in litterfall to a decreasing Mn availability, because of the increase in soil pH values and the absence of Mn in the fertilizer (Table S3; González Sarango et al., 2021; Godo & Reisenauer, 1980; Hayes et al., 2014; Pan et al., 2014). The amendment of fertilizer plus lime increased the fluxes of all considered elements, even of Mn, with litterfall (Table S5), because of the strong fertilization effect on litterfall production in spite of partly unchanged or in the case of Mn even decreased nutrient concentrations (González Sarango et al., 2021).

Biochar had significant positive effects on Ca and Zn concentrations in litterfall during the whole sampling period (Table S4). Increasing Zn concentrations in litterfall or leaf in response to biochar amendment are in line with the findings of Ali et al. (2019), Farooq et al. (2020) and Jatav et al. (2018) who observed an improved micro-nutrient supply of plants after the amendment of biochar. Similar lacking effects of biochar amendment on Cu and Na concentrations in corn straw were reported by Haider et al. (2017) and Bai et al. (2015). The few effects of biochar on nutrient concentrations in litterfall were in line with the reported effect of biochar on nutrient availability in soil assessed with the modified Olsen extract ( $\text{NaHCO}_3 + \text{EDTA}$ ) at the same study by González Sarango et al. (2022). Biochar did not influence any element flux, when the whole sampling period was considered (Table S5). Obviously, the input of all nutrients with the biochar except Ca and Zn was too small to improve the plant nutrient availability in soil (González Sarango et al., 2022). Our finding that biochar amendment even reduced the N concentration in litterfall is in line with findings of Akoto-Danso et al. (2019) and Gale et al. (2017). These authors reported that the N concentrations of the tissue of maize, amaranth and some herbaceous plants decreased after biochar amendment, likely because of microbial immobilization.

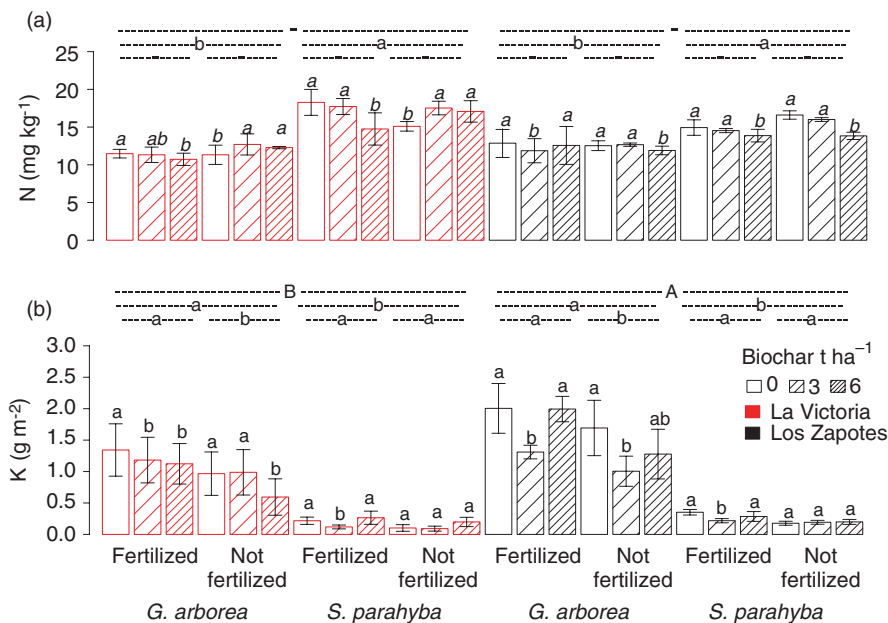
The fact that date had a significant effect on all element concentrations and fluxes with litterfall in our repeated measures ANOVA based on 4-mo aggregated values (Tables S4 and S5) led us to consider the dry and rainy seasons separately. The separate consideration of the element concentrations in litterfall and fluxes with

litterfall in the dry and rainy seasons revealed largely the same site, site block and tree species effects as already seen when considering the whole sampling period (Tables S6–S9). The effects of mineral fertilizer plus lime in each of the individual seasons were also similar to those observed for the whole sampling period. The significant increase of the Ca and Zn concentrations in the litterfall with biochar amendment was limited to the rainy season (Tables S6 and S8), during which biochar also increased Ca and Zn fluxes with litterfall (Figure 1, Table S7). For the dry season, we detected complex significant effects of biochar on N, Fe and Al concentrations in litterfall (Table S8). The amendment of biochar decreased the N concentrations in litterfall on the mineral fertilized plots at La Victoria and consistently on all plots at Los Zapotes marginally significantly. An exception was the 6 t ha<sup>-1</sup> biochar amendment to the mineral fertilized *G. arborea* plots (Figure 2a). On the not fertilized plots at La Victoria, biochar, in contrast, increased the N concentrations in litterfall during the dry season (Figure 2a). The application of 3 t ha<sup>-1</sup> of biochar decreased and that of 6 t ha<sup>-1</sup> increased Al and Fe concentrations in litterfall during the dry season (Table S8). Moreover, biochar decreased the K fluxes with litterfall during the dry season, albeit not consistently (Figure 2b). These results illustrate that particularly the biochar amendment of 6 t ha<sup>-1</sup> even decreased the supply of the macronutrients N and K during the dry season, possibly because it

increased N and K adsorption. In contrast, biochar had positive effects on the Ca and Zn supply during the rainy season and the Fe supply during the dry season (only in the 6 t ha<sup>-1</sup> of biochar treatments). The increase of the Al concentration in litterfall in response to the amendment of 6 t ha<sup>-1</sup> of biochar during the dry season reflects a potentially negative effect of biochar on plant performance. Overall, in our study, the effects of biochar on nutrient concentrations in litterfall and fluxes with litterfall were weak and partly even negative for plant performance in line with its weak effects on nutrient availability in soil (González Sarango et al., 2022). Similar to the findings of González Sarango et al. (2021) at the same study sites, de Farias et al. (2016) reported no response of native *Tachigali vulgaris* L.F.Gomes da Silva & H.C.Lima and exotic *Eucalyptus urophylla* × *Eucalyptus grandis* trees to the amendment of 2.5–20 vol% of biochar to the soil in a tree plantation in Mato Grosso, Brazil. Thus, even the much higher biochar amendment of de Farias et al. (2016) did not sufficiently improve the nutrient availability to the plantation trees. Santalla et al. (2011) even found that the charcoal included in mixed wood ash reduced the P and N availability in soil of a *Pinus radiata* D.Don plantation in northwest Spain. This is in line with our finding that the amendment of biochar to mineral fertilized plots even decreased N concentrations in litterfall during the dry season (Table S8). In contrast, Bélanger et al. (2004) concluded from a positive correlation between the charcoal

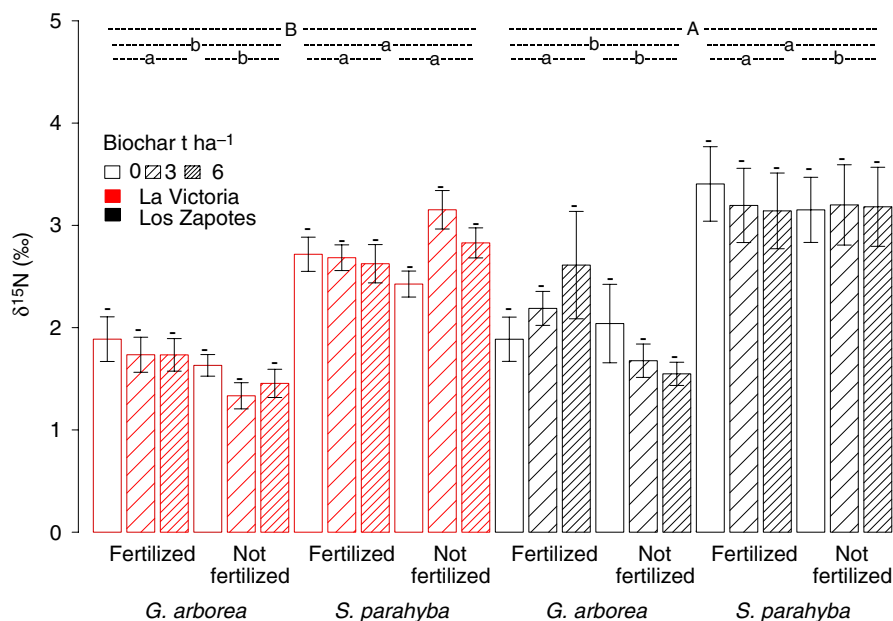


**FIGURE 1** Mean Ca (a) and Zn (b) fluxes with litterfall of the rainy season in all treatments of the afforestation experiment. Error bars show standard errors ( $n = 4$  at La Victoria and  $n = 3$  at los Zapotes). Different upper-case letters indicate significant differences between the study sites. Different lower-case letters in the uppermost row indicate significant differences between the tree species, in the middle row between fertilized and not fertilized plots and in the lowermost row among the different biochar amendment rates (in italics if marginally significant,  $p < 0.01$ ) according to an ANOVA followed by a Tukey's HSD post-hoc test at a significance level of  $p < 0.05$ .



**FIGURE 2** Mean N concentrations in litterfall (a) and K fluxes with litterfall (b) in the dry season in all treatments of the afforestation experiment. Error bars show standard errors ( $n = 4$  at La Victoria and  $n = 3$  at Los Zapotes). Different upper-case letters indicate significant differences between the study sites. Different lower-case letters in the uppermost row indicate significant differences between the tree species, in the middle row between fertilized and not fertilized plots and in the lowermost row among the different biochar amendment rates (in italics if marginally significant,  $p < 0.01$ ) according to an ANOVA followed by a Tukey's HSD post-hoc test at a significance level of  $p < 0.05$ .

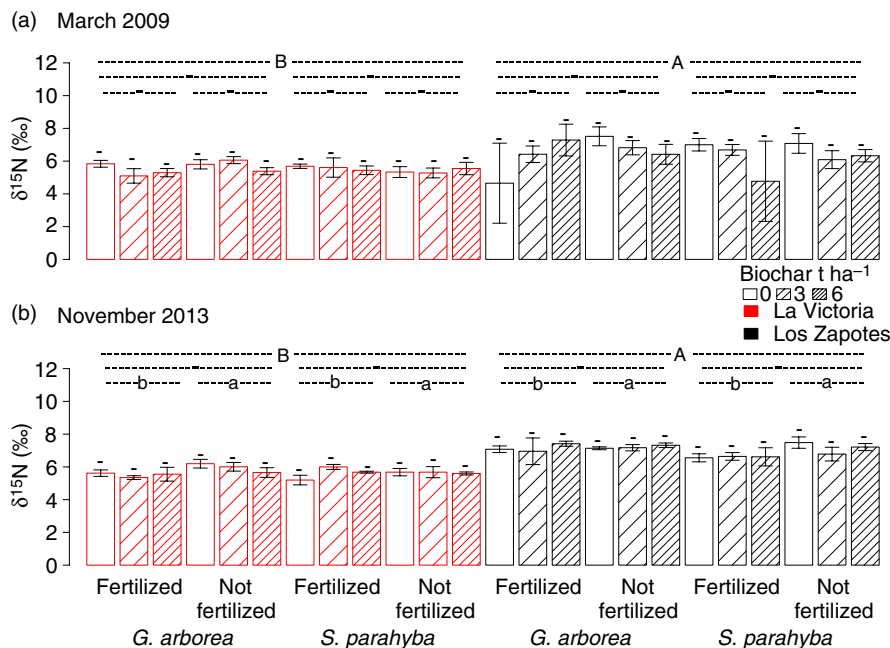
**FIGURE 3** Mean annual  $\delta^{15}\text{N}$  values in litterfall of all treatments at La Victoria and los Zapotes. Error bars show standard errors ( $n = 4$  at La Victoria and  $n = 3$  at los Zapotes). Different upper-case letters indicate significant differences between the study sites. Different lower-case letters in the uppermost row indicate significant differences between the tree species, in the middle row between fertilized and not fertilized plots and in the lowermost row between plots with and without biochar based on an ANOVA followed by a Tukey's HSD post-hoc test at a significance level of  $p < 0.05$ .



concentration in soil originating from a several decades old forest fire and the base cation concentrations in the forest floor of a mixed forest stand in Québec, Canada that charcoal can improve the base cation availability in the long term. The latter finding is only partly corroborated by our study where the Ca concentrations in litterfall and fluxes with litterfall increased in response to biochar amendment during the rainy season (Figure 1, Table S8).

### 3.2 | Stable N isotope ratios as indication of changed N cycling

Site, site block and tree species had significant effects on the  $\delta^{15}\text{N}$  values of litterfall (Table S4). The inherently more fertile site Los Zapotes (González Sarango et al., 2021), had significantly higher  $\delta^{15}\text{N}$  values in litterfall (Figure 3) and in soil (Figure 4). This is in line



**FIGURE 4** Mean  $\delta^{15}\text{N}$  values in mineral topsoils of all the treatments at La Victoria and Los Zapotes in March 2009 before the start of the experiment and in November 2013 at the end of the experiment. Error bars show standard errors ( $n = 4$  at La Victoria and  $n = 3$  at Los Zapotes). Different upper-case letters indicate significant differences between the study sites. Different lower-case letters in the uppermost row indicate significant differences between the tree species, in the middle row between fertilized and not fertilized plots and in the lowermost row between plots with and without biochar based on an ANOVA followed by a Tukey's HSD post-hoc test at a significance level of  $p < 0.05$ .

with the assumption that a higher N richness, as reflected by the higher mean annual N concentration of  $2.6 \pm 0.11 \text{ g kg}^{-1}$  and a lower C/N ratio of  $11 \pm 0.110$  in litterfall at Los Zapotes than at La Victoria ( $2.2 \pm 0.04 \text{ g kg}^{-1}$  and  $14 \pm 0.10$ ) resulted in a more open N cycle. The more open N cycle is reflected by higher  $\delta^{15}\text{N}$  values in soils and litterfall (Eshetu & Högberg, 2000; Hietz et al., 2011; Kleinebecker et al., 2014; Thomazo et al., 2011). The effect of site block might again be attributable to a higher nutrient richness of the soils at the lower than the upper slope, because of downslope-directed lateral nutrient transport, which had also been observed in a nearby native tropical montane forest (Wilcke et al., 2010). With increasing N richness, the  $\delta^{15}\text{N}$  values of litterfall are expected to increase. The significant influence of tree species on the  $\delta^{15}\text{N}$  values of litterfall seemed at the first glance to be related with the fact that *S. parahyba* is a legume which can fix  $\text{N}_2$  from the atmosphere via its symbiosis with *Rhizobia*. Surprisingly, the litterfall of *S. parahyba* had higher  $\delta^{15}\text{N}$  values than that of *G. arborea*, although we expected the reverse because  $\text{N}_2$  fixation should move the  $\delta^{15}\text{N}$  values of litterfall nearer to 0‰ (Hietz et al., 2011; Unkovich, 2013). We, therefore, suggest that *S. parahyba* did not or only to a limited extent fix  $\text{N}_2$  from the atmosphere, because of the N amendment in the fertilized and strong P limitation in the unfertilized treatments. *Gmelina arborea* showed a higher

litterfall, basal area and stem diameter than *S. parahyba* (González Sarango et al., 2021) and therefore likely took up more of the N fertilizer, which was urea with a  $\delta^{15}\text{N}$  value of  $-0.30\text{‰}$  than *S. parahyba*, resulting in the lower  $\delta^{15}\text{N}$  values of *G. arborea* than *S. parahyba* (Figure 3).

The amendment of fertilizer plus lime increased the  $\delta^{15}\text{N}$  values of litterfall significantly (Figure 3; Table S4), similar to the studies of Raymond et al. (2016) and Yan et al. (2020) which was more pronounced for *G. arborea* than *S. parahyba*. This is again unexpected because of the low  $\delta^{15}\text{N}$  value of the N fertilizer of  $-0.30\text{‰}$ . We can only speculate that the amendment of the complete mineral fertilizer plus lime improved the mineralization of soil organic matter and therefore also the supply of isotopically heavier N. A higher loss of isotopically light gaseous N after fertilization with urea may have additionally contributed to the higher  $\delta^{15}\text{N}$  values of the litterfall. Biochar did not influence the  $\delta^{15}\text{N}$  values of litterfall (Figure 3; Table S4).

The litterfall of *G. parahyba* showed lower  $\delta^{15}\text{N}$  values in the dry than in the rainy season, both in the plots with mineral fertilizer plus lime ( $1.14\text{--}2.9\text{‰}$  in the rainy season and  $1.00\text{--}2.53\text{‰}$  in the dry season) and without ( $1.09\text{--}3.52\text{‰}$ ,  $0.84\text{--}2.83\text{‰}$ ), irrespective of the amendment of biochar. We attribute this to reduced leaching of isotopically light N from the leaves during the dry season (Amundson et al., 2003; Carroll et al., 2016; Schwarz et al., 2011; Yan et al., 2020).



In March 2009 before the start of the experiment, the only significant difference in the  $\delta^{15}\text{N}$  values of the mineral topsoil existed between the two study sites. The more fertile site at Los Zapotes showed higher  $\delta^{15}\text{N}$  values than the site at La Victoria (Figure 4). This was likely attributable to the more open N cycle at Los Zapotes than at La Victoria with higher losses of isotopically light N. In November 2013 at the end of the experiment, the fertilization with N-isotopically light urea had slightly decreased the  $\delta^{15}\text{N}$  values of the topsoil. In contrast, neither the tree species nor the biochar amendment had significant effects on the  $\delta^{15}\text{N}$  values of the topsoil (Figure 4). The amendment of biochar did not influence the  $\delta^{15}\text{N}$  values of the topsoil, because the  $\delta^{15}\text{N}$  value of the biochar ( $6.36 \pm \text{SD } 0.80\text{‰}$ ) was similar to that of the topsoils ( $5.53 \pm 0.09\text{‰}$ ,  $n = 48$ , at La Victoria and  $6.37 \pm 0.23\text{‰}$ ,  $n = 36$ , at Los Zapotes). Our findings are in line with those of Reverchon et al. (2015) who reported that the addition of up to  $60 \text{ t ha}^{-1}$  of biochar did not change the  $\delta^{15}\text{N}$  values of the soil. Thus, the biochar did not significantly influence the soil N cycle.

## 4 | CONCLUSIONS

The amendment of up to  $6 \text{ t ha}^{-1}$  of biochar had a weak positive effect on Ca and Zn concentrations in litterfall and fluxes with litterfall restricted to the rainy season, partly supporting our first hypothesis for this season. However, for the dry season, our first hypothesis was not supported because biochar amendment even tended to decrease N concentrations in litterfall and decreased K fluxes with litterfall. The finding that the biochar did not influence the  $\delta^{15}\text{N}$  values of litterfall or soil with and without the simultaneous amendment of a complete mineral fertilizer plus lime suggested that it did not help close the N cycle. Thus, our second hypothesis is not supported. This underlines that the application of biochar at least at the rates chosen for our experiment did not substantially improve the nutrient economy of the two studied tree plantations in the Ecuadorian Amazonia.

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## CONFLICT OF INTEREST

The authors declare no conflict of interest.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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## REFERENCES

- Akoto-Danso, E. K., Manka'abusi, D., Steiner, C., Werner, S., Häring, V., Nyarko, G., Marschner, B., Drechsel, P., & Buerkert, A. (2019). Agronomic effects of biochar and wastewater irrigation in urban crop production of tamale, northern Ghana. *Nutrient Cycling in Agroecosystems*, *115*, 231–247. <https://doi.org/10.1007/s10705-018-9926-6>
- Ali, S., Rizwan, M., Noureen, S., Anwar, S., Ali, B., Naveed, M., Abd Allah, E. F., Alqarawi, A. A., & Ahmad, P. (2019). Combined use of biochar and zinc oxide nanoparticle foliar spray improved the plant growth and decreased the cadmium accumulation in rice (*Oryza sativa* L.) plant. *Environmental Science and Pollution Research*, *26*, 11288–11299. <https://doi.org/10.1007/s11356-019-04554-y>
- Amundson, R., Austin, A. T., Schuur, E. A. G., Yoo, K., Matzek, V., Kendall, C., Uebersax, A., Brenner, D., & Baisden, W. T. (2003). Global patterns of the isotopic composition of soil and plant nitrogen. *Global Biogeochemical Cycles*, *17*, 1031. <https://doi.org/10.1029/2002GB001903>
- Arif, M., Ilyas, M., Riaz, M., Ali, K., Shah, K., Ul Haq, I., & Fahad, S. (2017). Biochar improves phosphorus use efficiency of organic-inorganic fertilizers, maize-wheat productivity and soil quality in a low fertility alkaline soil. *Field Crops Research*, *214*, 25–37. <https://doi.org/10.1016/j.fcr.2017.08.018>
- Azeez, J. O. (2019). Recycling organic waste in managed tropical forest ecosystems: Effects of arboreal litter types on soil chemical properties in Abeokuta, southwestern Nigeria. *Forestry Research*, *30*, 1903–1911. <https://doi.org/10.1007/s11676-018-0753-z>
- Bai, S. H., Reverchon, F., Xu, C.-Y., Xu, Z., Blumfield, T. J., Zhao, H., van Zwieten, L., & Wallace, H. M. (2015). Wood biochar increases nitrogen retention in field settings mainly through abiotic processes. *Soil Biology and Biochemistry*, *90*, 232–240. <https://doi.org/10.1016/j.soilbio.2015.08.007>
- Barrow, C. (2012). Biochar: Potential for countering land degradation and for improving agriculture. *Applied Geography*, *34*, 21–28. <https://doi.org/10.1016/j.apgeog.2011.09.008>
- Bélanger, N., Côté, B., Fyles, J. W., Courchesne, F., & Hendershot, W. H. (2004). Forest regrowth as the controlling factor of soil nutrient availability 75 years after fire in a deciduous forest of southern Quebec. *Plant and Soil*, *262*, 363–272. <https://doi.org/10.1023/B:PLSO.0000037054.21561.85>

- Carroll, T. M., Thorp, J. H., & Roach, K. A. (2016). Autochthony in karst spring food webs. *Hydrobiologia*, 776, 173–191. <https://doi.org/10.1007/s10750-016-2750-6>
- Chan, K. Y., van Zwieten, L., Meszaros, I., Downie, A., & Joseph, S. (2008). Using poultry litter biochars as soil amendments. *Soil Research*, 46, 437–444. <https://doi.org/10.1071/SR08036>
- Choi, W.-J., Kwak, J.-H., Lim, S.-S., Park, H.-J., Chang, S. X., Lee, S.-M., Arshad, M. A., Yun, S.-I., & Kim, H.-Y. (2017). Synthetic fertilizer and livestock manure differently affect  $\delta^{15}\text{N}$  in the agricultural landscape: A review. *Agriculture, Ecosystems & Environment*, 237, 1–15. <https://doi.org/10.1016/j.agee.2016.12.020>
- Coplen, T. B., Hopple, J. A., Böhlke, J. K., Peiser, H. S., Rieder, S. E., Krouse, H. R., Rosman, K. J. R., Ding, T., Vocke, R. D., Jr., Revesz, K. M., Lamberty, A., Taylor, P., & De Bievre, P. (2002). *Compilation of minimum and maximum isotope ratios of selected elements in naturally occurring terrestrial materials and reagents*, Water-Resources Investigations Report. U.S. Geological Survey. <https://doi.org/10.3133/wri014222>
- Criscuoli, I., Alberti, G., Baronti, S., Favilli, F., Martinez, C., Calzolari, C., Pusceddu, E., Rumpel, C., Viola, R., & Miglietta, F. (2014). Carbon sequestration and fertility after centennial time scale incorporation of charcoal into soil. *PLoS One*, 9, e91114. <https://doi.org/10.1371/journal.pone.0091114>
- Dawes, M. A., Schleppei, P., Hättenschwiler, S., Rixen, C., & Hagedorn, F. (2017). Soil warming opens the nitrogen cycle at the alpine treeline. *Global Change Biology*, 23, 421–434. <https://doi.org/10.1111/gcb.13365>
- de Farias, J., Marimon, B. S., de Carvalho Ramos Silva, L., Petter, F. A., Andrade, F. R., Morandi, P. S., & Marimon-Junior, B. H. (2016). Survival and growth of native *Tachigali vulgaris* and exotic *Eucalyptus urophylla* × *Eucalyptus grandis* trees in degraded soils with biochar amendment in southern Amazonia. *Forest Ecology and Management*, 368, 173–182. <https://doi.org/10.1016/j.foreco.2016.03.022>
- De Mendiburu, F. (2017). *Agricolae: Statistical procedures for agricultural research*. R package version. <https://CRAN.R-project.org/package=agricolae>
- Deluca, T., Gundale, M., MacKenzie, M., & Jones, D. (2015). Chapter 14: Biochar effects on soil nutrient transformations. In J. Lehmann & S. Joseph (Eds.), *Biochar for environmental management science, technology, and implementation* (2nd ed., pp. 419–452). Routledge.
- Ding, Y., Liu, Y., Liu, S., Li, Z., Tan, X., Huang, X., Zeng, G., Zhou, L., & Zheng, B. (2016). Biochar to improve soil fertility. A review. *Agronomy for Sustainable Development*, 36, 5–18. <https://doi.org/10.1007/s13593-016-0372-z>
- Dormann, C. F., & Kühn, I. (2011). *Angewandte Statistik für die biologischen Wissenschaften*. 2. Aufl., Helmholtz-Zentrum für Umweltforschung - UFZ. [https://www.ufz.de/export/data/2/92353\\_deutschstatswork\\_23022011.pdf](https://www.ufz.de/export/data/2/92353_deutschstatswork_23022011.pdf)
- Drake, J. A., Carrucan, A., Jackson, W. R., Cavignaro, T. R., & Patti, A. F. (2015). Biochar application during reforestation alters species present and soil chemistry. *Science of the Total Environment*, 514, 359–365. <https://doi.org/10.1016/J.SCITOTENV.2015.02.012>
- El-Naggar, A., Lee, S. S., Rinklebe, J., Farooq, M., Song, H., Sarmah, A., Zimmerman, A., Ahmad, M., & Ok, Y. S. (2019). Biochar application to low fertility soils: A review of current status, and future prospects. *Geoderma*, 337, 536–554. <https://doi.org/10.1016/j.geoderma.2018.09.034>
- Eshetu, Z., & Högberg, P. (2000). Effects of land use on  $^{15}\text{N}$  natural abundance of soils in Ethiopian highlands. *Plant and Soil*, 222, 109–117. <https://doi.org/10.1023/A:1004777301260>
- Evans, R. D. (2001). Physiological mechanisms influencing plant nitrogen isotope composition. *Trends in Plant Science*, 6, 121–126. [https://doi.org/10.1016/S1360-1385\(01\)01889-1](https://doi.org/10.1016/S1360-1385(01)01889-1)
- Farooq, M., Ullah, A., Usman, M., & Siddique, K. H. M. (2020). Application of zinc and biochar help to mitigate cadmium stress in bread wheat raised from seeds with high intrinsic zinc. *Chemosphere*, 260, 127652. <https://doi.org/10.1016/j.chemosphere.2020.127652>
- Gale, N. V., Halim, M. A., Horsburgh, M., & Thomas, S. C. (2017). Comparative responses of early-successional plants to charcoal soil amendments. *Ecosphere*, 8, e01933. <https://doi.org/10.1002/ecs2.1933>
- Glaser, B., Balashov, E., Haumaier, L., Guggenberger, G., & Zech, W. (2000). Black carbon in density fractions of anthropogenic soils of the Brazilian Amazon region. *Organic Geochemistry*, 31, 669–678. [https://doi.org/10.1016/S0146-6380\(00\)00044-9](https://doi.org/10.1016/S0146-6380(00)00044-9)
- Glaser, B., Haumaier, L., Guggenberger, G., & Zech, W. (2001). The “Terra Preta” phenomenon: A model for sustainable agriculture in the humid tropics. *Naturwissenschaften*, 88, 37–41. <https://doi.org/10.1007/s001140000193>
- Glaser, B., Lehmann, J., & Zech, W. (2002). Ameliorating physical and chemical properties of highly weathered soils in the tropics with charcoal – a review. *Biology and Fertility of Soils*, 35, 219–230. <https://doi.org/10.1007/s00374-002-0466-4>
- Godo, G. H., & Reisenauer, H. M. (1980). Plant effects on soil manganese availability. *Soil Science Society of America Journal*, 44, 993–995. <https://doi.org/10.2136/sssaj1980.03615995004400050024x>
- González Sarango, E. M., Leimer, S., Valarezo Manosalvas, C. V., & Wilcke, W. (2022). Does biochar improve nutrient availability in Ultisols of tree plantations in the Ecuadorian Amazonia? *Soil Science Society of America Journal*, 86, 1072–1085. <https://doi.org/10.1002/saj2.20421>
- González Sarango, E. M., Valarezo Manosalvas, C., Mora, M., Villamagua, M. Á., & Wilcke, W. (2021). Biochar amendment did not influence the growth of two tree plantations on nutrient-depleted Ultisols in the south Ecuadorian Amazon region. *Soil Science Society of America Journal*, 85, 862–878. <https://doi.org/10.1002/saj2.20227>
- Gross, J., & Ligges, U. (2015). *nortest: Tests for Normality*. R package version (1.0-4). <https://CRAN.R-project.org/package=nortest>
- Haider, G., Steffens, D., Moser, G., Müller, C., & Kammann, C. I. (2017). Biochar reduced nitrate leaching and improved soil moisture content without yield improvements in a four-year field study. *Agriculture, Ecosystems and Environment*, 237, 80–94. <https://doi.org/10.1016/J.AGEE.2016.12.019>
- Hayes, P., Turner, B. L., Lambers, H., & Laliberté, E. (2014). Foliar nutrient concentrations and resorption efficiency in plants of contrasting nutrient-acquisition strategies along a 2-million-year dune chronosequence. *Ecology*, 102, 396–410. <https://doi.org/10.1111/1365-2745.12196>
- Hernandez-Soriano, M. C., Kerré, B., Goos, P., Hardy, B., Dufey, J., & Smolders, E. (2016). Long-term effect of biochar on the stabilization of recent carbon: Soils with historical inputs of charcoal. *GCB Bioenergy*, 8, 371–381. <https://doi.org/10.1111/gcbb.12250>
- Hietz, P., Turner, B. L., Wanek, W., Richter, A., Nock, C. A., & Wright, S. J. (2011). Long-term change in the nitrogen cycle of tropical

- forests. *Science*, 334, 664–666. <https://doi.org/10.1126/science.1211979>
- Högberg, P. (2008). Tansley review No. 95  $^{15}\text{N}$  natural abundance in soil-plant systems. *The New Phytologist*, 137, 179–203. <https://doi.org/10.1046/j.1469-8137.1997.00808.x>
- Homeier, J., Hertel, D., Camenzind, T., Cumbicus, N. L., Maraun, M., Martinson, G. O., Poma, L. N., Rillig, M. C., Sandmann, D., Scheu, S., Veldkamp, E., Wilcke, W., Wullaert, H., & Leuschner, C. (2012). Tropical Andean forests are highly susceptible to nutrient inputs – Rapid effects of experimental N and P addition to an Ecuadorian montane forest. *PLoS One*, 7, e47128. <https://doi.org/10.1371/journal.pone.0047128>
- Hu, P., Zhang, Y., Liu, L., Wang, X., Luan, X., Ma, X., Chu, P. K., Zhou, J., & Zhao, P. (2019). Biochar/struvite composite as a novel potential material for slow release of N and P. *Environmental Science and Pollution Research*, 26(17), 17152–17162. <https://doi.org/10.1007/s11356-019-04458-x>
- Ibell, P. T., Xu, Z., & Blumfield, T. J. (2013). The influence of weed control on foliar  $\delta^{15}\text{N}$ ,  $\delta^{13}\text{C}$  and tree growth in an 8-year-old exotic pine plantation of subtropical Australia. *Plant and Soil*, 369, 199–217. <https://doi.org/10.1007/s11104-012-1554-3>
- Inglett, P. W., Reddy, K. R., & McCormick, P. V. (2004). Periphyton chemistry and nitrogenase activity in a northern Everglades ecosystem. *Biogeochemistry*, 67, 213–233. <https://doi.org/10.1023/B:BIOG.0000015280.44760.9a>
- Jaramillo Botero, C., Santos, R. H. S., Fardim, M. P., Pontes, T. M., & Sarmiento, F. (2008). Produção de serapilheira e aporte de nutrientes de espécies arbóreas nativas em um sistema agroflorestal na Zona da Mata de Minas Gerais. *Árvore*, 32, 869–877. <https://doi.org/10.1590/S0100-676220080005000012>
- Jatav, H. S., Singh, S. K., Singh, Y., & Kumar, O. (2018). Biochar and sewage sludge application increases yield and micronutrient uptake in rice (*Oryza sativa* L.). *Communications in Soil Science and Plant Analysis*, 49, 1617–1628. <https://doi.org/10.1080/00103624.2018.1474900>
- Kellman, L. M., & Hillaire-Marcel, C. (2003). Evaluation of nitrogen isotopes as indicators of nitrate contamination sources in an agricultural watershed. *Agriculture, Ecosystems and Environment*, 95, 87–102. [https://doi.org/10.1016/S0167-8809\(02\)00168-8](https://doi.org/10.1016/S0167-8809(02)00168-8)
- Kishimoto, S., & Sugiura, G. (1985). Charcoal as a soil conditioner. Symposium on Forest products research. *International Achievements for the Future*, 5, 12–23.
- Kleinebecker, T., Hölzel, N., Prati, D., Schmitt, B., Fischer, M., & Klaus, V. H. (2014). Evidence from the real world:  $^{15}\text{N}$  natural abundances reveal enhanced nitrogen use at high plant diversity in central European grasslands. *Ecology*, 102, 456–465. <https://doi.org/10.1111/1365-2745.12202>
- Kookana, R., van Zwieten, L., Krull, E., & Singh, B. (2011). Biochar application to soil: Agronomic and environmental benefits and unintended consequences. *Advances in Agronomy*, 112, 103–143. <https://doi.org/10.1016/B978-0-12-385538-1.00003-2>
- Lehmann, J. (2007). Bio-energy in the black. *Frontiers in Ecology and the Environment*, 5, 381–387. [https://doi.org/10.1890/1540-9295\(2007\)5\[381:BITB\]2.0.CO;2](https://doi.org/10.1890/1540-9295(2007)5[381:BITB]2.0.CO;2)
- Lehmann, J., Gaunt, J., & Rondon, M. (2006). Bio-char sequestration in terrestrial ecosystems – A review. *Mitigation and Adaptation Strategies for Global Change*, 11, 403–427. <https://doi.org/10.1007/s11027-005-9006-5>
- Lehmann, J., Pereira da Silva, J., Steiner, C., Nehls, T., Zech, W., & Glaser, B. (2003). Nutrient availability and leaching in an archaeological Anthrosol and a Ferralsol of the Central Amazon basin: Fertilizer, manure and charcoal amendments. *Plant and Soil*, 249, 343–357. <https://doi.org/10.1023/A:1022833116184>
- Lemon, J. (2006). Plotrix: A package in the red light district of R. *R-News*, 6, 8–12.
- Levia, D. F., & Frost, E. E. (2006). Variability of throughfall volume and solute inputs in wooded ecosystems. *Progress in Physical Geography*, 30, 605–632. <https://doi.org/10.1177/0309133306071145>
- Likens, G. E. (2013). *Nutrient cycles and mass balances. In Biogeochemistry of a Forested Ecosystem* (pp. 139–161). Springer. [https://doi.org/10.1007/978-1-4614-7810-2\\_6](https://doi.org/10.1007/978-1-4614-7810-2_6)
- Lima, H. N., Schaefer, C. E., Mello, J. W., Gilkes, R. J., & Ker, J. C. (2002). Pedogenesis and pre-Columbian land use of “Terra Preta Anthrosols” (“Indian black earth”) of Western Amazonia. *Geoderma*, 110, 1–17. [https://doi.org/10.1016/S00162-7061\(02\)00141-6](https://doi.org/10.1016/S00162-7061(02)00141-6)
- Lin, Z., Liu, Q., Liu, G., Cowie, A. L., Bei, Q., Liu, B., Wang, X., Ma, J., Zhu, J., & Xie, Z. (2017). Effects of different biochars on *Pinus elliottii* growth, N use efficiency, soil  $\text{N}_2\text{O}$  and  $\text{CH}_4$  emissions and C storage in a subtropical area of China. *Pedosphere*, 27, 248–261. [https://doi.org/10.1016/S1002-0160\(17\)60314-X](https://doi.org/10.1016/S1002-0160(17)60314-X)
- Major, J., Rondon, M., Molina, D., Riha, S. J., & Lehmann, J. (2010). Maize yield and nutrition during 4 years after biochar application to a Colombian savanna oxisol. *Plant and Soil*, 333, 117–128. <https://doi.org/10.1007/s11104-010-0327-0>
- Mandal, S., Thangarajan, R., Bolan, N. S., Sarkar, B., Khan, N., Ok, Y. S., & Naidu, R. (2016). Biochar-induced concomitant decrease in ammonia volatilization and increase in nitrogen use efficiency by wheat. *Chemosphere*, 142, 120–127. <https://doi.org/10.1016/j.chemosphere.2015.04.086>
- Nelissen, V., Rütting, T., Huygens, D., Staelens, J., Ruyschaert, G., & Boeckx, P. (2012). Maize biochars accelerate short-term soil nitrogen dynamics in a loamy sand soil. *Soil Biology and Biochemistry*, 55, 20–27. <https://doi.org/10.1016/j.soilbio.2012.05.019>
- Novotny, E. H., Maia, C. M. B. d. F., Carvalho, M. T. d. M., & Madari, B. E. (2015). Biochar: Pyrogenic carbon for agricultural use - a critical review. *Ciência do Solo*, 39, 321–344. <https://doi.org/10.1590/01006683rbc20140818>
- Pan, Y., Koopmans, G. F., Bonten, L. T. C., Song, J., Luo, Y., Temminghoff, E. J. M., & Comans, R. N. J. (2014). Influence of pH on the redox chemistry of metal (hydr)oxides and organic matter in paddy soils. *Soils and Sediments*, 14, 1713–1726. <https://doi.org/10.1007/s11368-014-0919-z>
- Pardo, L. H., Templer, P. H., Goodale, C. L., Duke, S., Groffman, P. M., Adams, M. B., Boeckx, P., Boggs, J., Campbell, J., Colman, B., Compton, J., Emmett, B., Gundersen, P., Kjønaas, J., Lovett, G., Mack, M., Magill, A., Mbila, M., Mitchell, M. J., ... Wessel, W. (2006). Regional assessment of N saturation using foliar and root  $\delta^{15}\text{N}$ . *Biogeochemistry*, 80, 143–171. <https://doi.org/10.1007/s10533-006-9015-9>
- Park, H.-J., Lim, S.-S., Kwak, J.-H., Baek, W.-J., Yoon, K.-S., Choi, S.-M., & Choi, W.-J. (2015). Nitrogen inputs with different substrate quality modified pH, Eh, and N dynamics of a paddy soil incubated under waterlogged conditions. *Communications in Soil Science and Plant Analysis*, 46, 2234–2248. <https://doi.org/10.1080/00103624.2015.1069324>
- Pratiwi, E. P. A., Hillary, A. K., Fukuda, T., & Shinogi, Y. (2016). The effects of rice husk char on ammonium, nitrate and phosphate

- retention and leaching in loamy soil. *Geoderma*, 277, 61–68. <https://doi.org/10.1016/J.GEODERMA.2016.05.006>
- Raymond, J., Fox, T., & Strahm, B. (2016). Understanding the fate of applied nitrogen in pine plantations of the southeastern United States using  $^{15}\text{N}$  enriched fertilizers. *Forests*, 7, 270. <https://doi.org/10.3390/f7110270>
- Reverchon, F., Bai, S. H., Liu, X., & Blumfield, T. J. (2015). Tree plantation systems influence nitrogen retention and the abundance of nitrogen functional genes in the Solomon Islands. *Frontiers in Microbiology*, 6, 1–12. <https://doi.org/10.3389/fmicb.2015.01439>
- Santalla, M., Omil, B., Rodríguez-Soalleiro, R., & Merino, A. (2011). Effectiveness of wood ash containing charcoal as a fertilizer for a forest plantation in a temperate region. *Plant and Soil*, 346, 63–78. <https://doi.org/10.1007/s11104-011-0794-y>
- Schwarz, M. T., Oelmann, Y., & Wilcke, W. (2011). Stable N isotope composition of nitrate reflects N transformations during the passage of water through a montane rain forest in Ecuador. *Biogeochemistry*, 102, 195–208. <https://doi.org/10.1007/s10533-010-9434-5>
- Sebilo, M., Billen, G., Mayer, B., Billiou, D., Grably, M., Garnier, J., & Mariotti, A. (2006). Assessing nitrification and denitrification in the Seine River and estuary using chemical and isotopic techniques. *Ecosystems*, 9, 564–577. <https://doi.org/10.1007/s10021-006-0151-9>
- Shi, W., Ju, Y., Bian, R., Li, L., Joseph, S., Mitchell, D. R. G., Munroe, P., Taherymoosavi, S., & Pan, G. (2020). Biochar bound urea boosts plant growth and reduces nitrogen leaching. *Science of the Total Environment*, 701, 134424. <https://doi.org/10.1016/j.scitotenv.2019.134424>
- Silva, A. K. L., Vasconcelos, S. S., de Carvalho, C. J. R., & Cordeiro, I. M. C. C. (2011). Litter dynamics and fine root production in *Schizolobium parahyba* var. *amazonicum* plantations and re-growth forest in eastern Amazon. *Plant and Soil*, 347, 377–386. <https://doi.org/10.1007/s11104-011-0857-0>
- Song, X., Pan, G., Zhang, C., Zhang, L., & Wang, H. (2016). Effects of biochar application on fluxes of three biogenic greenhouse gases: A meta-analysis. *Ecosystem Health and Sustainability*, 2, 1–13. <https://doi.org/10.1002/ehs2.1202>
- Swamy, S. L., Kushwaha, S. K., & Puri, S. (2004). Tree growth, biomass, allometry and nutrient distribution in *Gmelina arborea* stands grown in red lateritic soils of Central India. *Biomass and Bioenergy*, 26, 305–317. <https://doi.org/10.1016/j.biombioe.2003.08.007>
- Thomazo, C., Ader, M., & Philippot, P. (2011). Extreme  $^{15}\text{N}$ -enrichments in 2.72-Gyr-old sediments: Evidence for a turning point in the nitrogen cycle. *Geobiology*, 9, 107–120.
- Unkovich, M. (2013). Isotope discrimination provides new insight into biological nitrogen fixation. *The New Phytologist*, 198, 643–646. <https://www.jstor.org/stable/newphytologist.198.3.643>
- Velescu, A., Homeier, J., Bendix, J., Valarezo, C., & Wilcke, W. (2021). Response of water-bound fluxes of potassium, calcium, magnesium and sodium to nutrient additions in an Ecuadorian tropical montane forest. *Forest Ecology and Management*, 501, 119661. <https://doi.org/10.1016/j.foreco.2021.119661>
- Vigulu, V., Blumfield, T. J., Reverchon, F., Bai, S. H., & Xu, Z. (2019). Nitrogen and carbon cycling associated with litterfall production in monoculture teak and mixed species teak and *Flueggea* stands. *Soils and Sediments*, 19, 1672–1684. <https://doi.org/10.1007/s11368-019-02275-w>
- Warnes, G., Bolker, B., Bonebakker, L., Gentleman, R., Huber, W., Liaw, A., Lumley, T., Maechler, M., Magnusson, A., Moeller, S., Schwartz, M., & Venables, B. (2009). *Gplots: Various R programming tools for plotting data*. R package version. <https://github.com/talgalili/gplots>
- Wilcke, W., Boy, J., Goller, R., Fleischbein, K., Valarezo, C., & Zech, W. (2010). Chapter 43. Effect of topography on soil fertility and water flow in an Ecuadorian lower montane forest. In L. A. Bruijnzeel, F. N. Scatena, & L. S. Hamilton (Eds.), *Tropical montane cloud forests: Science for conservation and management* (pp. 402–409). Cambridge University Press.
- Wilcke, W., Yasin, S., Abramowski, U., Valarezo, C., & Zech, W. (2002). Nutrient storage and turnover in organic layers under tropical montane rain forest in Ecuador. *Soil Science*, 53, 15–27. <https://doi.org/10.1046/j.1365-2389.2002.00411.x>
- Wilcke, W., Yasin, S., Valarezo, C., & Zech, W. (2001). Change in water quality during the passage through a tropical montane rain forest in Ecuador. *Biogeochemistry*, 55, 45–72. <https://doi.org/10.1023/A:1010631407270>
- Wullaert, H., Homeier, J., Valarezo, C., & Wilcke, W. (2010). Response of the N and P cycles of an old-growth montane forest in Ecuador to experimental low-level N and P amendments. *Forest Ecology and Management*, 260, 1434–1445. <https://doi.org/10.1016/j.foreco.2010.07.021>
- Xia, H., Riaz, M., Zhang, M., Liu, B., El-Desouki, Z., & Jiang, C. (2020). Biochar increases nitrogen use efficiency of maize by relieving aluminum toxicity and improving soil quality in acidic soil. *Ecotoxicology and Environmental Safety*, 196, 110531. <https://doi.org/10.1016/j.ecoenv.2020.110531>
- Yan, G., Han, S., Zhou, M., Sun, W., Huang, B., Wang, H., Xing, Y., & Wang, Q. (2020). Variations in the natural  $^{13}\text{C}$  and  $^{15}\text{N}$  abundance of plants and soils under long-term N addition and precipitation reduction: Interpretation of C and N dynamics. *Forest Ecosystems*, 7, 49. <https://doi.org/10.1186/s40663-020-00257-w>
- Zhang, R., Zhang, Y., Song, L., Song, X., Hänninen, H., & Wu, J. (2017). Biochar enhances nut quality of *Torreya grandis* and soil fertility under simulated nitrogen deposition. *Forest Ecology and Management*, 391, 321–329. <https://doi.org/10.1016/J.FORECO.2017.02.036>

## SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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