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Nutrient recycling using biochar on plinthosol of Burkina Faso : a soil-plant column experiment

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LUCIE BLONDIAU

TRAVAIL DE FIN D'ÉTUDES PRÉSENTÉ DANS LE CADRE DE L'OBTENTION DU DIPLÔME
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Abbreviations

°C	Degree Celsius
AAS	Atomic absorption spectrometry
ADE	Amazonian Dark Earth
AV1	One factor analysis of variance
BC	Pristine biochar amendment
BD	Bulk density
BUNASOL	Bureau National des Sols (Burkina Faso)
C	Compost amendment
C_{org}	Organic carbon
Ca	Calcium
CBC	Co-compost amendment
CEC	Cation exchange capacity
Corg	Organic carbon
DAP	Diammonium phosphate
DW	Dry Weight
EDTA	Ethylenediaminetetraacetic acid
K	Potassium
KM	Kjeldhal method
MBC	Mineral loaded biochar amendment
Mg	Magnesium
MLB	Mineral loaded biochar
N	Nitrogen
N-fertilizers	Nitrogenous fertilizer
NH_3	Ammonia
NH_4^+	Ammonium
NPK	Nitrogen phosphorus potassium fertiliser
OC	Organic carbon
OM	Organic matter
P	Phosphorus
PB	Pristine biochar
pH	Potential of hydrogen
PVC	Polyvinyl Chloride
rpm	Rotation per minute
SRF	Slow release fertilizer
T	Control treatment
UNB	Nazy Boni university
w/w	Weight to weight ratio
WRB	World reference base

Abstract

Most soils in Burkina Faso are highly weathered and have low nutrients and organic matter contents. Hence, organic and mineral fertilisers are necessary to maintain the nutrients balance and crops productivity. However, because of high leaching rates during wet seasons, plants do not efficiently uptake these fertilisers. Biochar has been heralded as a solution for nutrients retention and soil quality improvement. Despite low initial nutrients concentrations, woody biochar nutrients properties can be boosted by combination with nutrients-rich compounds that entails nutrients releasing for longer time period in the soil. Therefore, nutrients loaded biochar could serve as a new slow release fertiliser in West Africa.

This work studied two enhanced biochar: compost activated (CBC) and NPK loaded (MBC) biochar and focused on their effects on nutrients recycling in a plinthosol from Burkina Faso in a soil column experiment.

Five treatments have been studied: co-composted biochar (CBC), compost (C), pristine biochar (BC), NPK loaded biochar (MBC) and reference columns (T). The cycling of nutrients under these different treatments has been analysed through the study of soil columns leachates and plants minerals uptakes.

The results showed no significant differences between the shoot yields and the plants nutrients uptake. In terms of initial stock, CBC and BC treatments allowed better cations retention and yet, did not reduce phosphorus (P) leaching. On the contrary, MBC decreased P leaching but not cations losses.

This study set the first explanatory steps to understand the effects of nutrients loaded cotton biochar in plinthosols and how this new technology could be used effectively to solve soils fertility problems encountered in West Africa.

Keywords: Burkina Faso, Plinthosol, co-composted biochar, mineral loaded biochar, nutrient cycling, soil columns experiment

Résumé

Les sols du Burkina Faso sont pauvres en nutriments et en matières organiques. L'application d'engrais minéraux et organiques est indispensable pour assurer la fertilité des sols et le maintien de des rendements. De plus, les fortes précipitations lors des saisons des pluies lessivent ces fertilisants, les rendant indisponibles pour les cultures. Un amendement du sol en biochar pourrait améliorer la rétention des intrants. Le biochar produit à partir des matériaux ligneux à une faible teneur en nutriments. Cependant, combiné à des matériaux plus riches en éléments nutritifs, ce dernier peut être enrichi et libérer des nutriments plus longtemps dans le sol. Ce biochar pourrait être utilisé comme nouvel amendement *slow release* en Afrique de l'Ouest.

Cette étude porte sur l'effet du biochar, enrichi par incorporation dans le processus de compostage, ou par mélange avec une solution d'engrais NPK, sur le recyclage des nutriments dans un Plinthosol du sud-ouest du Burkina Faso.

Dans cette expérimentation en colonnes de sols, cinq modalités ont été considérées : du co-compost (CBC), du compost (C), du biochar (BC) et du biochar macéré avec de l'engrais NPK (MBC) ainsi que des colonnes témoins (T). Les percolas et les biomasses récoltés ont été analysés afin de quantifier leurs concentrations en nutriments.

Les rendements et l'assimilation des nutriments par les plantes n'ont pas été significativement impactés par les traitements étudiés. Une rétention plus importante des cations a été observée pour CBC mais couplée à une augmentation de la perte de phosphore. À l'inverse, MBC a permis de réduire les pertes de P mais n'a pas empêché le lessivage des cations.

Cette étude peut être considérée comme une première approche dynamique de l'influence des biochars activés sur des sols burkinabés et d'évaluer comment cette technologie pourrait être utilisée pour améliorer la fertilité des sols en Afrique de l'Ouest.

Mots clés: Burkina Faso, Plinthosol, co-compost, mineral loading, cycle des nutriments, colonnes de sol

Part 1: Article

1. Introduction

The decline of soil fertility in Sub-Saharan Africa has become a major concern for scientists and governments (Gray and Morant, 2003). The depletion of major nutrients such as nitrogen (N) and phosphorous (P) from soils and the low organic matter content constitute the main bottleneck to an increased agricultural productivity (Schlecht *et al.*, 2006). This observation is all the more alarming considering the current rise of population growth and the urgent need to increase food supplies in West Africa (Scoones and Toulmin, 1999).

Burkina Faso is amongst the nations with the highest population growth rate on earth and yet is still ranked 183rd on the HDI scale in 2014 (FAO, 2014). Also the low resilience of this country to economical and environmental crisis implies a high vulnerability for 85% of the population depending upon agricultural sector as a source of income (Etongo *et al.*, 2018).

Soils of Burkina Faso need recurring organic and inorganic fertilisers applications to maintain soils fertility and productivity (Tojo Soler *et al.*, 2011). However, the socio-economical constraints led to an intensification of agricultural practices without restoration of the soils nutrient balance and induced soil nutrient depletion (Bationo, Lompo and Koala, 1998; Tojo Soler *et al.*, 2011). Furthermore, heavy rainfalls occurring during the wet season intensify soil degradation and nutrients leaching. Considering the actual rising price of fertilisers, relying exclusively on mineral fertilisers to restore soils nutrients stocks in Burkina Faso is economically unsustainable for small producers (Agegnehu, Srivastava and Bird, 2017). In addition, excessive fertiliser application threatens the environment. For example excess of nitrogenous fertilisers (N-fertiliser) are responsible for groundwater pollution and eutrophication (Kanthle *et al.*, 2016; Zhang *et al.*, 2016).

Biochar is a product inspired from the Amazonian Dark Earth (ADE) also called *terra preta* and has recently drawn the scientific community's attention for its various properties. Biochar is produced by thermal treatment in oxygen deficient conditions of organic matter (OM), e.g. pyrolysis, at low temperature and is intended to be used to amend soil (Fischer and Glasser, 2013; Kuppusamy et al, 2016). Its characteristics and their influence on the soil environnment vary mainly depending on the biochar feedstock and the pyrolysis conditions. Biochars made from lignocellulosic materials have a lower content in nutrients than nutrients rich feedstock such as manure. In contrast, woody biochars have a better structure and are more appropriate for modification of soil physical properties modification (Khan *et al.*, 2008; Cantrell *et al.*, 2012; Jeffery *et al.*, 2017). However, most studies agree that its highly porous matrix, inherent tortuosity and ability to sorb cations are key elements for retaining nutrients and water in the soil (Mukherjee and Andrew R. Zimmerman, 2013; Barnes *et al.*, 2014).

Furthermore, biochar is composed of stable carbon and has a high carbon sequestration potential compared to straw or manure (Lehmann and Joseph, 2009; Qayyum *et al.*, 2014). Thus, black carbon might be a solution to enrich soils carbon content in regions with a high turnover of OM (Jeffery *et al.*, 2017). Theoretically biochar amendment has a high potential for improving soil fertility and mitigating climate change (Woolf *et al.*, 2010; Scholz *et al.*, 2014).

However, as of 2016, still 75% of biochar experiments were still unsuccessful in increasing crop yields without adding any fertiliser or organic manure (Kuppusamy et al, 2016). Despite this observation, biochar is known to be an efficient nutrient carrier (Agegnehu, Srivastava and Bird, 2017) and could be used to produce a new generation of slow realease fertilisers (SRF) when combined to nutrient-rich components such as chemical or organic fertilisers (Schmidt *et al.*, 2017; Gwenzi *et al.*, 2018). Enriched biochar might gradually release nutrients in the soil and meet the plants needs over a longer time period while reducing nutrients leaching from cultivated areas.

Furthermore a synergy between biochar and other OM has been observed during the composting process (Fischer and Glaser, 2013). On the one hand, biochar is acting as an active bulking agent maintening the aeration and the structure of the compost pile (Steiner *et al*., 2011) whilst providing a profitable habitat for microorganisms (Lehmann *et al*., 2011). On the other, the temperature increase during the composting process speeds up the oxidation of the biochar particles and increases its adsorption capacity (Schmidt *et al*., 2015; Khan *et al*., 2016). Fisher and Glaser (2013) also reported that biochar decreases nitrogen and water losses in compost, and reduces greenhouse gas emissions. In other words, biochar technology might be seen as an attempt to optimize the organic matter recycling by getting it back to the field and limiting the carbon mineralisation.

Although numerous studies have dealt with the effect of biochar with or without fertilisers combination, to our knowledge none concern cotton stalks as feedstock material on highly weathered soils of South West Burkina Faso nor compare mineral and organic activated biochar simultanuously. This study aims to quantify the effects of co-composted biochar and biochar loaded with NPK fertiliser on the nutrients cycling in a soil-plant system trough a columns experiment.

2. Material and methods

2.1 Experimental site description

The soil was sampled near the village of Koumbia (11°14'27.4"N 3°42'28.2"W; 290 m alt.) located in South-West Burkina Faso. The tropical weather of this area is defined as a Sudanian climate (Akoudjin *et al.*, 2016) composed of a wet season occurring from May/June to September which includes almost all the yearly rainfalls with an average of 740 mm and a dry season starting from September to May. The total annual rainfalls and mean temperature range respectively around 900 mm and 27.7°C.

The soil was sampled in February 2018 from a field being usually intended for maize cultivation and never amended with biochar. The sandy loam soil was shallow with a lateritic endurement at 20 cm deep and developing on a granitic parent material (Burgeon, 2017). It was defined as Plinthosol according to the World Reference Base (WRB) (FAO and SSSB, 2015) and was referred as FLIPP on the map of the Bureau National des Sols (BUNASOL) of Burkina Faso (Annex 2). The zone was flat on a large area.

2.2 Soil sampling

Seventy-five kilogrammes of soil were randomly sampled in the crop ridges at three different points of the field and air-dried. The bulk density (BD) of the soil was measured both in the ridges and the furrows of the field with metal rings of 250 cm^3 according to the BIOSE AxeESP LBO01 protocol (Losseau, 2013). The soil was watered before inserting the ring in the ground in order to conserve its sandy structure. The BD rings samples were dried for 48 hours in a proofer at 40°C and weighed.

2.3 Biochar production, co-composting and mineral loading

The biochar production was undertaken at the Nazi Boni University (UNB) in Bobo-Dioulasso (Burkina Faso). It was produced from cotton straws in a Kon-Tiki flame curtain pyrolyser between 650 and 700°C (Burgeon, 2017) in June 2017. The biochar was stored outdoors before being oven-dried (40°C) in February 2018.

Two composts, with (co-compost) and without (compost) biochar, were implemented in Gembloux Agro-Bio Tech (Belgium) according to the Berkley rapid composting method (Das *et al.*, 2011; Maqbool and Rehman, 2015). Two bioreactors were built using plastic containers of 70 L. The containers internal wall was covered with insulation sheets in order to minimize the heat loss caused by the small volumes of the composts. Small holes were pierced at the bottom of the containers to allow aeration of the bottom of the pile and the evacuation of the compost leachate. The composts with or without biochar were made with fresh (60% of total volume) and 6 months old (20% of total volume) cattle manure and biochar (20% of total volume) or straws (20% of total volume). Those materials were manually chopped in smaller pieces, between 2 and 5 cm long, to allow for a faster and easier breakdown of the organic matter and were applied layer by layer in the container. Both composts were placed in a greenhouse maintained at 25°C during the entire process. The compost piles were turned over for the first time 5 days after starting the composts and then every 2 or 3 days. The temperature was recorded hourly with a temperature data logger (LASCAR® electronics, EL-

USB-1) placed in the centre of the compost pile and collected through the EasyLog® software (Figure 16).

Biochar was also enriched by combination with NPK fertiliser. To do so, a mixture 50g/50g (w/w ratio) of dried biochar and NPK fertiliser (14:23:14) was mixed up with 600 ml of deionized water (volume equal to the considered biochar volume). The mixture was heated until the total dissolution of the NPK granules and macerated for 48 hours. The mixture was then filtered with Whatman® folded filters (595 ½) and the biochar was oven dried (40°C).

2.4 Soil columns preparation

Soil columns were made with polyvinyl chloride (PVC) tubes 30 cm in length and 10 cm in diameter. A column stopper was stuck with all-use glue and PVC glue, and pierced with a central 1.2 cm hole. The columns inside wall was softly grounded to decrease the risks of sidewall effect by increasing the wall roughness. A lay flat hose was used for leachate evacuation that was collected in 250 ml plastic flasks. A draining layer was made with geotextile, Argex® marbles and coarse decarbonized sand at the bottom of the column (**Annexe 1**.a).

Over this layer, 20 cm of soil was dry packed with a pestle at a bulk density ranging between 1.5 g/cm^3 and 1.7 g/cm^3, which correspond to the bulk density of the ridges and the furrow measured in the sampled field (cf.-2.2. Soil sampling). Light grooves were drawn between the different soil additions to ensure a better consolidation of the entire column.

In the top 10 cm of the column the soil was mixed with the tested amendment grinded at 2 mm: pristine biochar (BC), mineral loaded biochar (MBC), co-compost (CBC) and compost (C). The application ratios refer to the ones currently applied on maize fields in Burkina Faso. More specifically the MBC application ratio comes from doses applied in microfertigation and directly applied at the seedling base. In that case, it was hypothesized that the seed hole was equivalent to the top 10 cm of the column. Control columns (T) were made with non-amended soil. The columns were disposed following a completely randomized design with 5 treatments and 4 replicates (**Annexe 1**.b). The quantities and proportions are summarized in the Table 1.

Table 1: Treatment used in the experiment (co-comopost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and control (T)) and their associated abbreviation (abbrv.), hectare equivalent application and amendment weight to soil weight ratio n=4.

Abbrv.	Treatment	Replicates	Application ratio	Weight ratio
CBC	Co-compost	4	10 T/ha	0.59% (w/w)
C	Compost	4	10T/ha	0.59% (w/w)
BC	Biochar	4	10 T/ha	0.59% (w/w)
MBC	Mineral loaded biochar	4	4 g/seed hole	0.32 % (w/w)
T	Control	4	Non amended	Non amended

Before sowing the columns were saturated with distilled water. After the drainage of the columns, 3.5 g of ryegrass (*Lolium perenne* L.) seeds were sown on the top of the columns. The columns were watered every 3 days with 150 ml of distilled water to simulate the amount of precipitation during the rainy season in Burkina Faso.

A NPK fertiliser 14-23-14 was applied at a rate of 150 kg-N/ha, 246 kg-P/ha and 150 kg-K/ha (Bumane, 2009) on day 18th of the experimentation. Eventually, every column received 120 mg of N, 200 mg of P and 120 mg of K. A total of 15 waterings were done during the 40 days experiment. However to limit the number of analyses of the soil solution without losing the nutrients leaching rate overview some leachates were grouped progressively before and after the fertiliser pulse (Table 2).

Forty days after sowing, the aboveground biomass was harvested and dry weighted (DW) after 72 hours in the proofer.

In order to analyse relative data, i.e. independent from the initial nutrients stock of each treatment, the amount of nutrients assimilated by the plants or leached out the column were divided by the initial nutrients stocks of each treatments. Those ratios will be used in the discussion section.

Table 2: Time table and analyses groups as a function of times. "Days" corresponds to the day when the columns were leached, with the associated leaching name in the row "leaching". The row "Groups" correspond to the number of different leaching episodes analysed as one.

Days	0	1	4	7	10	13	16	19	22	25	28	31	33	36	39
Leaching	L0	L1	L2	L3	L4	L5	L6	L7	L8	L9	L10	L11	L12	L13	L14
Groups	S1	S2	S3		S4			S5	S6	S7		S8			

NPK fertiliser addition

2.5 Chemicals analyses

The initial materials (NPK, pristine biochar and mineral loaded biochar) were analysed for nutrients content: N_{tot}, P_{tot}, K_{tot}, Ca_{tot} and Mg_{tot}. N_{tot} was quantified using the Kjeldhal method (KM) with a direct titration by hydrochloric acid (HCl) 1M or 0,1M. P_{tot} was quantified after a mineralisation by perchloric acid ($HClO_4$) by spectrophotometry with molybdenum blue colouring reagent at 880 nm. The other elements were analysed by atomic absorption spectrometry (AAS) after a triacid dissolution (HF, HClO4 and HCl). The pH_w and pH_{KCl} of both biochars were measured after 2 hours of shaking and 10 minutes centrifugation at 3000 rpm with a ratio 1:10 (w/v) solution extract (Cayuela *et al.*, 2013) of distilled water or potassium chloride (KCl).

In addition the pH of the soils from the different treatments were measured following the same protocol but with a ratio of 2:5 (w/v).

The following soils chemical properties (N_{tot}, C_{tot}, K_{av}, P_{av}, C_{av}, Mg_{av}, pH_{KCl} and CEC) were determined by the laboratory of la Hulpe. K_{av}, P_{av}, C_{av}, Mg_{av} were extracted with ammonium acetate 0.5 N and

EDTA 0.02 M at pH 4.65 (Lakanen and Ervio, 1971). Then, P was analysed by spectrophotometry with molybdenum blue coloration at 880 nm and K, Ca and Mg were analysed by AAS according to the procedure described in CWEA S-II-12. Organic carbon and total nitrogen contents were obtained by dry combustion respectively according to a method derived from ISO 10694 and ISO 13878. The soil CEC were quantified according to a method derived from ISO 23470 with hexamminecobalt trichloride solution as extractant. The soil pH_{KCl} were measured conforming to NF ISO 10390 with a solution of KCl 1M in a ratio 1:5 (v/v).

Analyses of compost and co-compost were carried out by the laboratory of Michamps. The C_{org} was quantified by loss on ignition at 550 °C and N_{tot} was analysed by KM as explained above. The NH_4^+ concentration was quantified by steam distillation on fresh matter. Available K, Mg, Ca, and P were quantified by AAS and spectrophotometry after calcination at 450°C.

In addition, P, K, Mg and Ca concentrations in the soil leachates were quantified. Spectrophotometry was used for P quantification while atomic absorption spectrophotometry was used for K, Mg and Ca. Ammonium was also quantified through titration of NH_4^+ with HCl (0,01M) after steam distillation with 50 ml of sodium hydroxide (NaOH - 35 %) and collected in 25 ml of boric acid (H_3BO_4 -4%).

Nutrients concentrations were determined in the ryegrass plants. The nutrients mineralomass was quantified by multiplying the concentration of nutrients by ryegrass biomass. Before analyses, the biomass was cut in 1cm^2 pieces. Nitric acid (HNO3 65%) and perchlorid acid (HClO4 70%) were added to the cut plants for mineralisation and then dissolved in HCl (10%). Ca, K and Mg were then quantified using the same methods described above. P was quantified with ammonium molybdate tetrahydrates colouring reagent by spectrophotometry at 430 nm.

2.6 Statistical analyses

One-way variance analyses (AV1) were performed with Minitab® 18 software and applied ($\alpha = 0,05$) after equal variance testing. Tukey's honestly significant difference (Tukey's HSD) test was used to distinguish the significant differences among treatments. Regression lines were drawn with Microsoft®Excel.

3. Results

3.1. Chemical properties of the untreated soil and amended soils

The studied soil (T) sampled in Koumbia (Table 3) has typical properties of highly weathered tropical soils that are characterized by low CEC and carbon contents. Moreover, the chemical analyses showed that the studied soil had low bioavailable nutrients concentrations.

Amended soils have slightly higher Mg_{av}, Ca_{av} and C_{org} concentrations than the control while K and P contents are two to ten folds higher ($p\text{-value} < 0.001$) than the control (T) (Table 3). In opposition neither N contents nor topsoil CEC were significantly influenced by the treatments.

The soils pH_w values ranged between 6,5 and 7,3 and increased as follows T < MBC < BC < CBC <C.

Table 4 shows the chemical properties of the pristine biochar and the NPK fertiliser used for biochar mineral loading as well as the ones of the maceration product. Surprisingly the analyses of the NPK fertiliser showed that the announced ratio of 14:23:14 was not respected by the supplier. K and P concentrations were respectively higher and lower than expected quantities. The NPK loaded biochar gained 790 % of N, 907% of P, 733% of K, 184% of Mg compared to the initial pristine bioachar but lost 22% of Ca. The pH_w value of pristine biochar and mineral loaded biochar were respectively of 9.7 and 6.7.

The compost and the co-compost have similar chemical properties as shown in Table 5.

Table 3: Chemical properties of top layer of the columns (0-10 cm) with co-composted amendment (CBC), compost amendment (C), pristine biochar amendment (BC), mineral loaded biochar amendment (MBC) and control (T) . The table shows the means ± std. Tukey's HSD test results are shown by the letters. Means that do not share the same letter are significantly different (p-value<0.05).

	CBC	C	BC	MBC	T
P_{av} [mg/100g]	2.34±0.02 b	2.91±0.01 b	0.60±0.02 c	8.13±0.58 a	0.32±0.05 c
K_{av} [mg/100g]	20.2±0.7 b	34.8±1.5 a	11.1±0.6 d	25.9±1.7 c	3.8±0.3 e
Mg_{av} [mg/100g]	4.40±0.17 a	4.42±0.08 a	3.59±0.07 b	3.58±0.16 b	2.99±0.06 c
Ca_{av} [mg/100g]	35.7±3.0 a	36.5±2.2 a	34.2±3.4 ab	29.1±3.0 ab	27.6±3.6 b
Humus [%]	1.17±0.03 b	1.20±0.02 b	1.57±0.09 a	0.99±0.05 c	0.74±0.06 d
N [%]	0.029±0.001 a	0.035±0.002 a	0.026±0.002 a	0.031±0.006 a	0.028±0.003 a
CEC [$cmol_c$/kg]	0.81±0.71 a	1.09±0.28 a	0.50±0.40 a	0.21±0.12 a	0.08±0.08 a
pH_w	7.2±0.1 a	7.3±0.0 a	7.2±0.1 a	6.6±0.1 b	6.5±0.1 b
pH_{KCl}	6.3±0.0 a	6.2±0.1 a	6.4±0.1 a	5.7±0.1 b	5.3±0.14 c

Table 4: Initial properties of the biochar, the mineral loaded biochar and the NPK (14:23:14) used for this maceration.

	N [%]	P [mg/100g]	K [mg/100g]	Mg [mg/100g]	Ca [mg/100g]
Pristine Biochar	0.54 ± 0.02	386 ± 40	1395 ± 141	339 ± 22	2542 ± 27
NPK Loaded Biochar	4.27 ± 0.03	3507 ± 86	10239 ± 501	625 ± 40	1988 ± 260
NPK fertiliser	13.56 ± 0.13	9192 ± 250	23111 ± 2070	616 ± 27	1124 ± 56

Table 5: Compost and co-compost chemical properties quantified on fresh biomass except the pH_w and pH_{KCl} that were quantified on dry matter.

	Corg %	N tot [kg/T fresh]	N-NH4 %N	K [kg/T fresh]	P [kg/T fresh]	Mg [kg/T fresh]	Ca [kg/T fresh]	pHw	pH_{KCl}
Co--compost	40	3.49	0.9	10	2.5	1.04	3.74	9.95	9.46
Compost	38.5	4.68	1.1	13.14	3.35	1.42	4.5	9.95	9.43

3.2 Nutrient leaching

For all treatments, Ca (figure 1) and Mg (figure 2) concentrations showed similar trends after the first leaching. The Ca first leaching concentrations increased significantly as follows $\mathrm{T_c{<}BC_b{<}CBC_a{<}C_a{<}MBC_a}$ depending on the column treatment. The Mg first leaching concentration increased significantly as follow: $\mathrm{T_c{<}BC_b{<}CBC_b{<}C_{ab}{<}MBC_a}$. The letters in index represent the groups formed by the Tukey's HSD test. Treatments that do not share the same letters are significantly differents (p-value < 0.05). Both elements concentrations in soil leachates decreased rapidly during the 4 first leachings and then levelled out showing no more significant differences (p-value > 0.05) between the treatments. No influence of the fertiliser pulse (F-pulse) was noticed after day 18. The total leached quantities of Ca and Mg are significantly different (p-value < 0.001) and ranged respectively from 16.7 to 107.9 mg per column and from 5.1 to 31.2 mg per column.

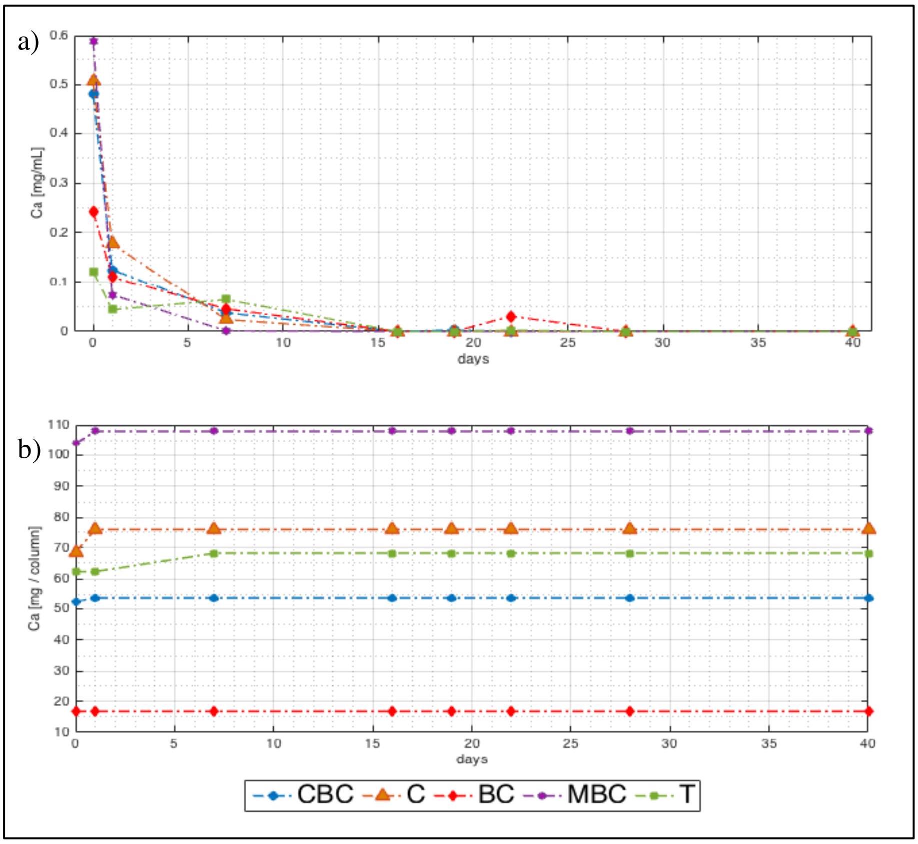


Figure 1: Evolution of Ca concentrations in leachate over time (a) and Ca cumulated quantities leached over time (b) for the column amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and non-amended (T).

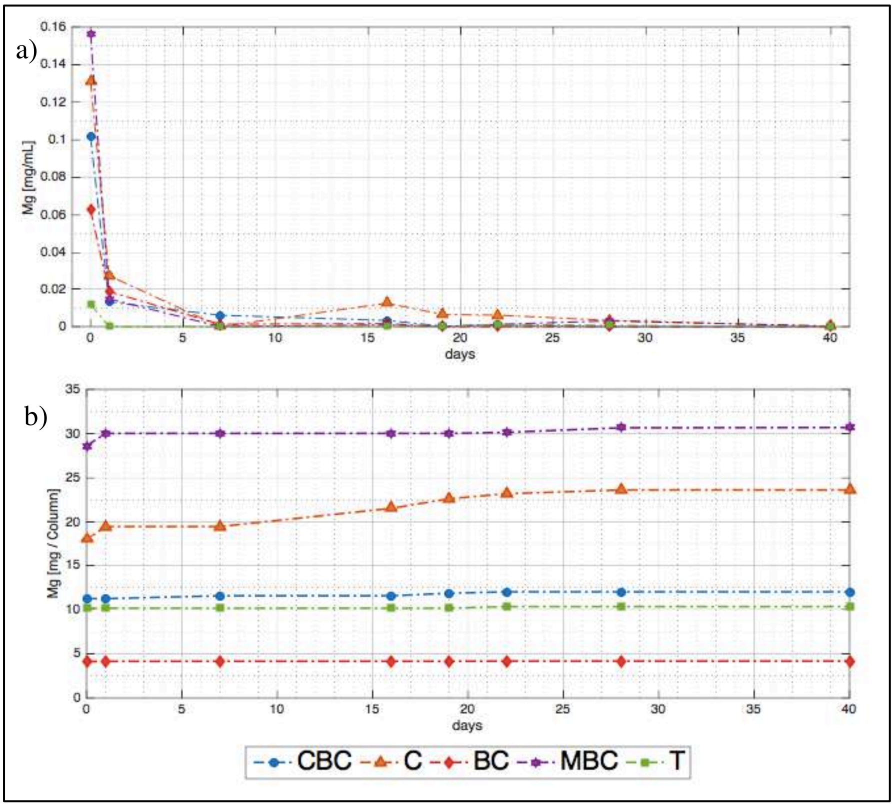


Figure 2: Evolution of Mg concentrations in leachate over time (a) and Mg cumulated quantities leached over time (b) for the column amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and non-amended (T).

The K first leaching concentrations (Figure 3) increased as follow T<CBC<MBC<BC<C and all means were equal (p-value > 0.05). K concentrations decreased rapidly after the first four waterings. The fertiliser addition induced a well-marked rise of the K concentrations with significant differences between the treatments (p-value < 0.05). After F-pulse, the C treatment decreased more steadily than the CBC, BC and T treatments. The MBC concentration levelled out before to decrease on day 28. The total K amounts leached through the columns were significantly different between the treatments (p-value < 0.05) and ranged from 37.7 to 323.6 mg per column.

Before the F-pulse, P concentrations (Figure 4) were below the detection limit (0.1 mg/L) and NH_4 concentrations (figure 5) were low except for the MBC treatment. After day 18, the fertiliser addition induces a sharp increase of the concentration and an immediate decrease on day 22 except for the MBC treatment. On the one hand the NH_4^+ concentration of MBC did not decrease as fast and as linearly as the other modalities. On the other hand, MBC P concentration peak after the fertiliser addition is delayed by one watering in comparison with the treatments concentrations.

The P soil leachate concentrations between the treatments were not significantly different during the entire experiment (p-value > 0.05).

Concerning NH_4^+ leachings, the MBC treatment concentration was always significantly higher (p-value < 0.001) than the other one, except on day 19 and 22 where means were all equals between the modalities.

The total amounts of percolated P (p-value > 0.05) and NH_4^+ (p-value < 0.001) varied respectively from 0.3 to 10.6 mg per column and from 25.5 to 165.2 mg per column.

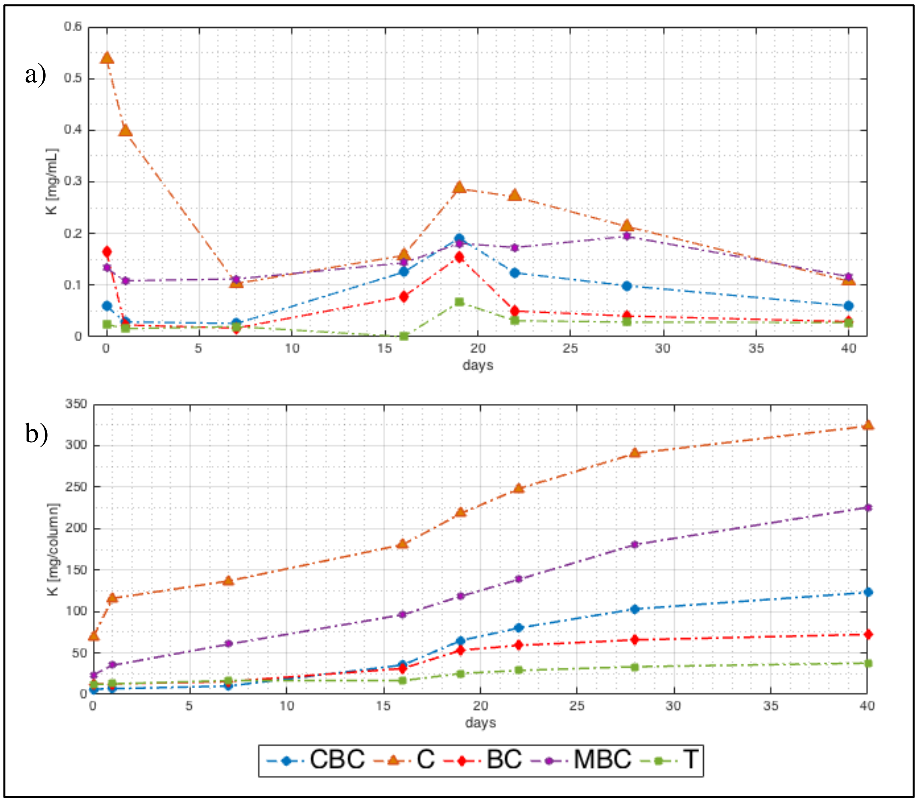


Figure 3: Evolution of K concentrations in leachate over time (a) and K cumulated quantities leached over time (b) for the column amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and non-amended (T).

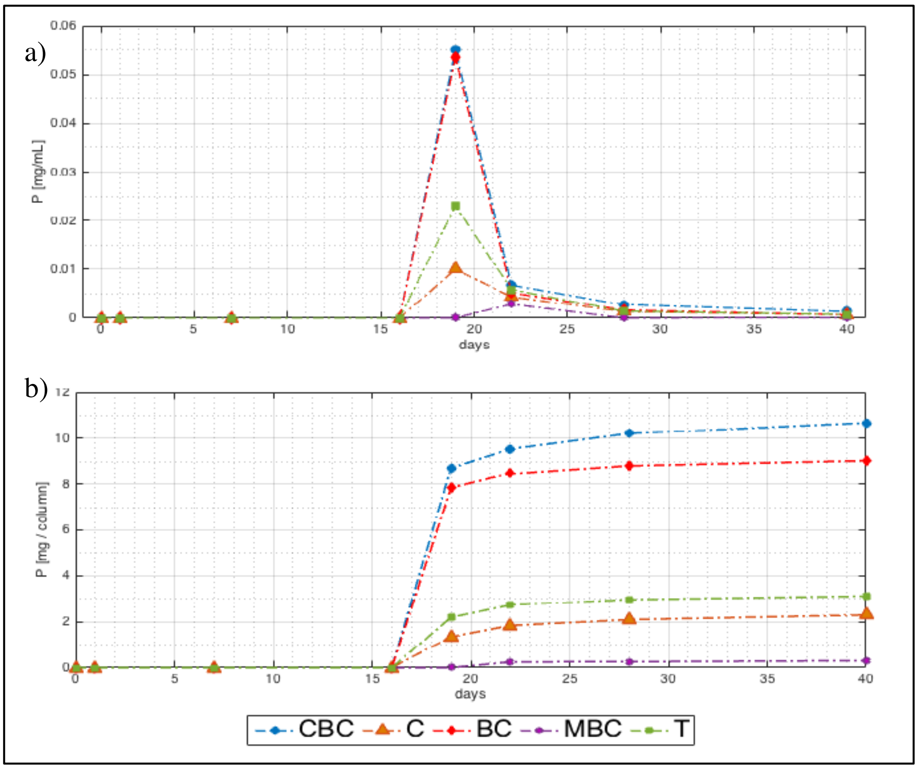


Figure 4: Evolution of P concentrations in leachate over time (a) and P cumulated quantities leached over time (b) for the column amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and non-amended (T).

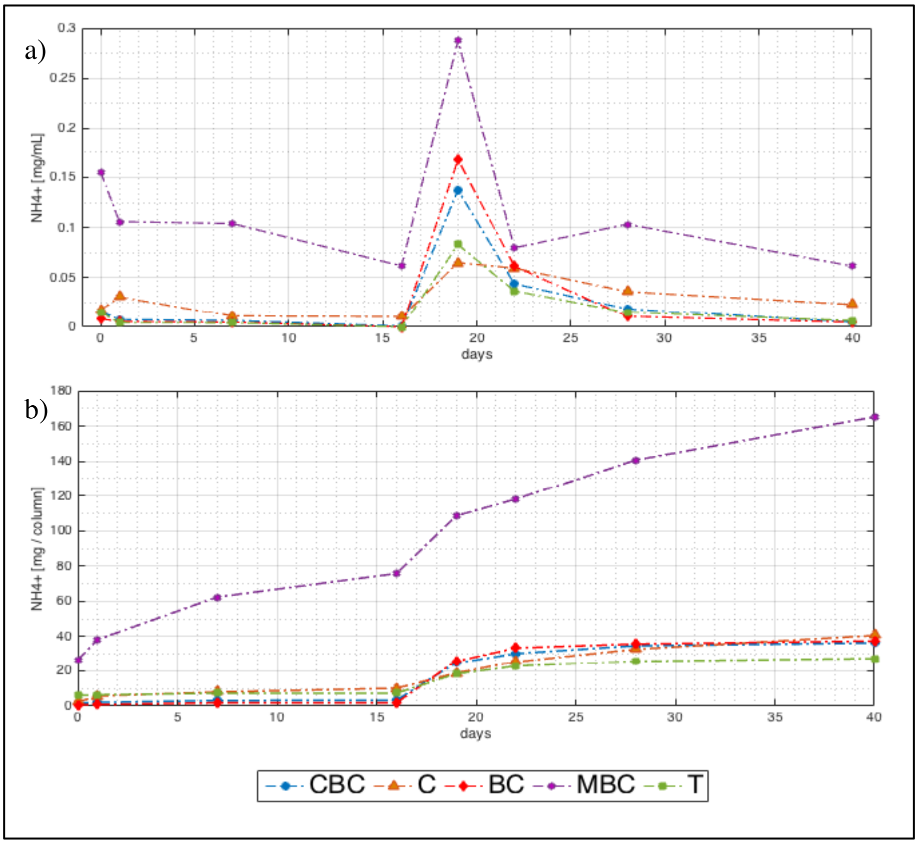


Figure 5: Evolution of NH_4^+ concentrations in leachate over time (a) and NH_4^+ cumulated quantities leached over time (b) for the column amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and non-amended (T).

3.3 Ryegrass biomass and nutrients uptake

The treatments means of the biomass shoot yields were equals according to Tukey's HSD test and the ANOVA test (p-value = 0.224) meaning that no significant observation can be drawn from Figure 6. However slight tendencies have been noticed amongst the treatments.

CBC and MBC biomass weight were respectively 76% and 51% lower than the control column (Figure 6). In opposite the ryegrass yield of the BC treatment was enhanced by 58%. The plants shoot weight of treatment C is similar to the control.

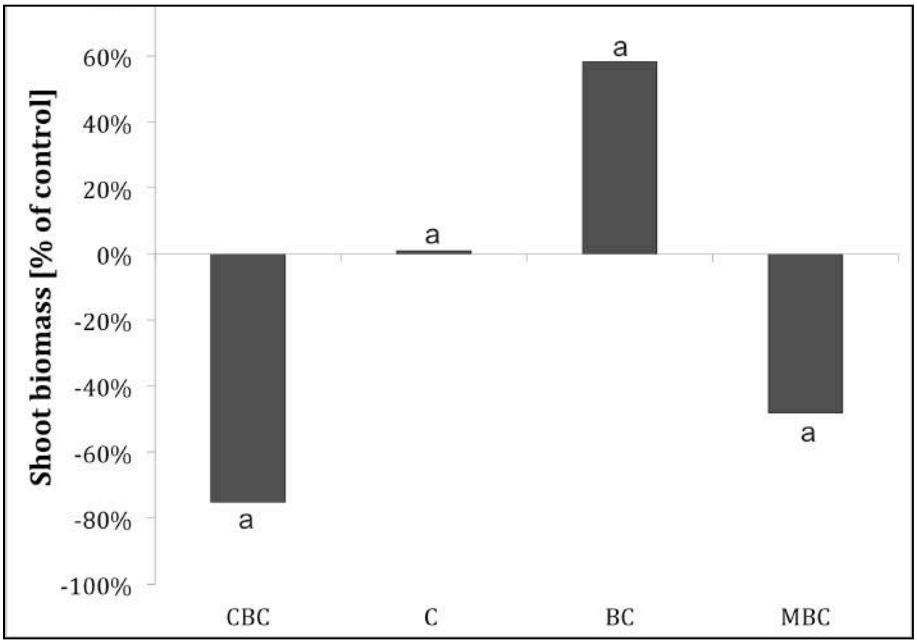


Figure 6: Shoot yields of the harvested ryegrass in ratio of the control yield for the soils amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC). The letters represent Tukey's HSD test. The columns means that do not share the same letter are significantly different (p-value < 0.05).

Figure 7 shows P, K, Mg and Ca shoot uptakes. Similarly than in the biomass weight case, none of the treatments means were significantly different (p-value > 0.05). Still, trends between the treatments can be analysed

Plants from CBC treatments systematically assimilated fewer nutrients than the others and varied from 65 and 87% lower than the control. MBC also had a negative impact on the uptake but ranging from 25 % to 73 % of T.

In opposition BC has a high positive impact on the biomass and on the nutrients uptake, from 10% to 104% higher than T. Although the C treatment shows a positive P uptake of 33% compared to the non-amended columns, it has negative impact on the Ca, Mg and K nutrient uptake from -9% to -47% compare to T.

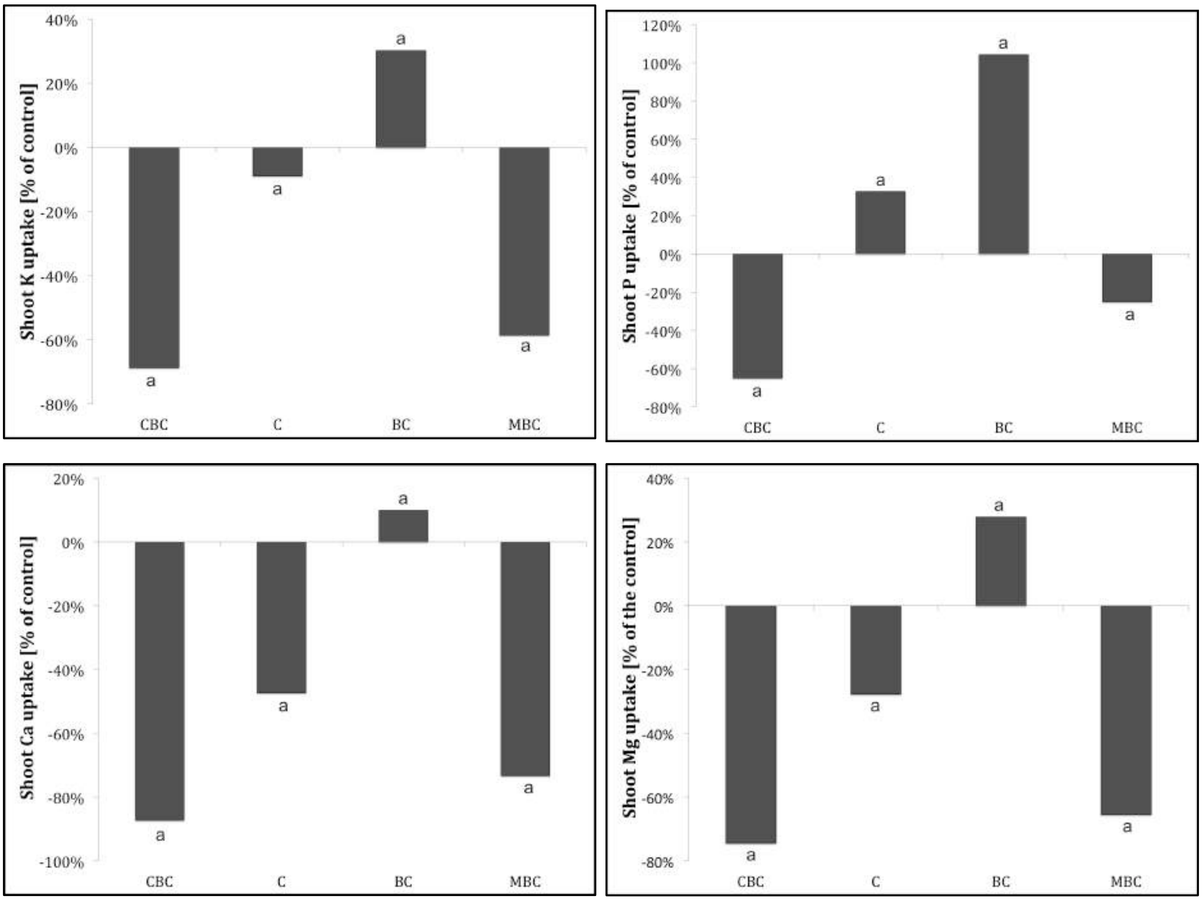


Figure 7: Shoot yields of the harvested ryegrass in ratio of the control yield for the soils amended with co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC). The letters represent Tukey's HSD test. The columns means that do not share the same letter are significantly different (p-value < 0.05).

4. Discussion

4.1 Impact of co-composting and NPK maceration on biochar properties

This work studies two different processes for biochar loading and enhancement: co-composting and maceration in NPK fertiliser solution. Both of them influenced the properties of the initial pristine biochar.

Enhancing the nutrients concentration in biochar by maceration in NPK was highly efficient in terms of K_{tot} (+733%), Mg_{tot} (+184%), N_{tot} (+790%) and P_{tot} (+907%) concentrations (Table 4). Different mechanisms can explain this nutrient enrichment as pore diffusion trough concentrations gradients, fixation on biochar functional groups, Vanderwaals forces and coprecipitation (Seymour and Bourdon, 2003; Wang and Chen, 2006; Berber-Mendoza *et al.*, 2018; Gwenzi *et al.*, 2018).

Higher P concentration on the biochar is less likely explained by adsorption since most biochar binding sites are negatives (Kammann *et al.*, 2015). Conte *et al.* (2013) and Kammann *et al.* (2015) reported that anions such as hydrated phosphate (PO_4^-) and nitrate (NO_3^-) were bound by unusual hydrogen-water bounds with the biochar pores.

However, the biochar contained a lower Ca concentration after maceration than the pristine biochar (Table 4). This is most likely due to the Ca concentration differential between the solid biochar (2542±27 mg/100g) and the NPK liquid phase (1124 ± 56 mg/100g). Indeed, this discrepancy created a gradient that led to Ca desorption from the biochar pores and surface in order to establish the solution Ca equilibrium (Singer and Munns, 1991; Seymour and Bourdon, 2003).

In addition this desorption might have benefit the adsorption of other cations on the newly vacant sites. For instance Ca and Mg are both bivalent cations with larger hydrated radius, respectively 0.96 nm and 1.08 nm (Weil and Brady, 2017). The Ca displacement might have contributed to Mg adsorption on the biochar surface vacant sites. Referring to this first finding, concentrations of the maceration solution must be managed adequately to enhance the biochar quality without altering its inherent nutrients resources.

The pH of the NPK loaded biochar was 3 units lower than the pristine biochar. Ahmad *et al.* (2018) observed the same phenomenon after loading biochar with NPK fertiliser. This was attributed to the loss of basic cations during the maceration and filtration of the mixture. In addition, the diammonium phosphate (DAP) fertiliser could also caused the acidification of biochar (Li *et al.*, 2014).

The co-compost did not show different characteristics than the compost (Table 5). Indeed, higher nutrients rates and pH in the co-composted product could have been expected because of the high water retention of the biochar and inputs of basic salts from biochar (Dias *et al.*, 2010; Singh, Singh and Cowie, 2010). Both composts presented particularly low NH_4 concentrations that ranged from 0.03 to 0.05 g/kg of fresh matter while the average cattle manure concentration for compost ranged from 0.3 to 2.0 g/kg of fresh mater (Bernal, Alburquerque and Moral, 2009). The high pH of 9.9 units of the co-compost and the compost might be an explanation since volatilisation of ammonia (NH_3) occurs for pH values over 7.5 (Bernal, Alburquerque and Moral, 2009). Furthermore frequent turnovers of the pile might have contributed to the gaseous NH_3 transfer from the compost pile to the atmosphere (Parkinson *et al.*, 2004).

However the N_{tot} concentration in the compost still fits the range from 4.2 to 8.1 N g/kg of fresh weight given by Bernal *et al.* (2009) but is slightly lower in the co-compost. There was no recuperation of the compost leachate during the composting and, thus, part of the N_{tot} might have been washed out the pile. Furthermore, the nitrate (NO_3^-) content of the pile was not monitored, nitrification of NH_4^+ might explain its low concentration in the compost.

In addition the compost did not follow the temperature evolution usually observed during the composting process. Indeed, a temperature rise is expected when the composting starts (i.e. thermophilic phase) (Insam and de Bertoldi, 2007). However in this study, the average temperature of both composts remained stable at around 29 °C. This last observation might have impacted the compost quality (Barral and Paradelo, 2011) and the biochar ageing (Khan *et al.*, 2016).

Yet, the frequent turnovers and the moisture of the pile ensured the contact and mixing between manure organic matter and the biochar particles and may have increased the biochar nutrients concentrations and have accelerated formation of new functional groups on the biochar surface (Cheng, Lehmann and Engelhard, 2008; Khan *et al.*, 2016).

4.2 Impact of amendment on soil properties

The initial soil pH was directly influenced by the amendment application: higher when biochar (BC) or compost (CBC and C) were added and lower after the application of mineral loaded biochar.

Genot *et al.* (2007) gave concentrations thresholds to characterize the nutrient status of soils depending on texture and CEC (K, Mg and Ca) (Table 6) or texture and pH_{KCl} (P) (Table 7). In this case the soil texture was considered as light due to the sandy loam texture of the soil.

The initial soil showed low concentrations of bioavailable P, Ca, Mg and K according to Genot *et al.* (2007) thresholds (Table 3).

Amended soils were classified for Mg and K respectively as good and very high (Table 6). However Ca concentrations remained low with a slight increase for the amended treatments compared to the control (Table 6). Similarly the P concentrations remained low with slight concentrations increase between the modalities except for the MBC treatment (Table 7). The NPK loaded biochar amended soil was classified as a soil with good P nutrient status. It is interesting to notice that the amendments increased significantly the concentration of K and Mg although the amendment to soil ratio (w/w) was lower than the 2% (w/w) ratio usually found in the literature (Schulz, Dunst and Glaser, 2013; Kammann *et al.*, 2015).

Still, according to Genot *et al.* (2007), C_{org} concentrations below 1.1g per 100g in sandy soils are considered poor in organic matter. However, all amendments increased the C_{org} concentrations from 0.37 to 0.78 g per 100g of dry matter. Thus, none of the treatments had a suitable carbon concentration. Likewise, no significant increase in CEC was noticed among the treatments (Table 3).

Those last two findings are surprisingly contrasting with major results in the literature where biochar addition increased the C soils contents (Steiner, 2003; Scholz *et al.*, 2014; Hernandez-soriano *et al.*, 2016). In addition, biochar was often reported to increase the soil CEC (Glaser *et al.*, 2001; Liang *et al.*, 2006). However, Burgeon (2017) found no CEC augmentation in a field study in Burkina Faso using a similar cotton biochar. Although some fresh biochars do not have high CEC, it increased over time with biochar surface oxidation and negative functional groups formation. (Cheng, Lehmann and Engelhard, 2008).

However, co-composted biochar generally gains in CEC during the thermophilic phase (Prost *et al.*, 2013) and the microorganisms action speed up the biochar surface ageing (Fischer and Glaser, 2013; Khan *et al.*, 2016). Yet, as mentioned above, temperatures increases were not observed. Thus, our composts might be more assimilated to the incubation product of biochar and organic matter as it has been studied in few works. Khan *et al*. (2016) found out that a biochar–compost incubation of 133 days did increase the nutrients content of the biochar and the surface carboxyl functional groups but did not enhance the mixture CEC. Besides compost and co-compost were produced over a short time period (24 days) while most existing works about co-composted biochar used compost made over more than 2.5 months (Steiner *et al*., 2011; Schmidt *et al*., 2014; Qayyum *et al*., 2017).

Table 6: Threshold values defined by Genot *et al*. (2007) for a light soil texture and for CEC below 5 cmolc/kg.

CEC [cmol$_c$/kg]	**Level**	**Ca [mg/100g]**	**Mg [mg/100g]**	**K [mg/100g]**
<5	Very high	> 92.2	> 6.5	> 10.4
	High	69.0 - 92.0	4.9 - 6.5	7.8 - 10.4
	Good	46.0 - 68.9	3.2 - 4.6	5.2 - 7.7
	Low	23.0 - 45.9	1.6 - 3.1	2.6 - 5.1
	Very low	< 23.0	< 1.6	< 2.6

Table 7: Threshold values defined by Genot *et al*. (2007) for a light soil texture and depending on the pH$_{KCl}$ of the soils.

	P [mg/100g]	
pH KCl	<5.5	≥ 5.5
high	> 9.0	>10.0
good	5.1 -9.0	6.6-10.0
low	<5.1	<6.6

4.3. Impact of biochar activation on nutrients dynamic in the soil-plant system

In this present section of the discussion, the leaching to stock ratio and the plant uptake to stock ratio will be used in order to rationalise the nutrients distribution with respect to the initial concentrations present in the studied soils figure 8.

However the initial stocks were extrapolated from the bioavailable concentrations using EDTA extractant and directly available nutrients concentrations might be overestimated. Therefore, this information is important to bear in mind because it has a direct influence on the ratios used along the following interpretations.

The regression line between shoot nutrients uptakes quantities and the leached nutrients quantities showed that there were low correlations between those mass balance components ($R^2 < 0.15$). This observation highlights the fact that lower uptakes were not only due to higher leaching and must imply other retention mechanisms in the soil. Since those results will not be used in this discussion, they are displayed in Part-2, 5 statistical analyses.

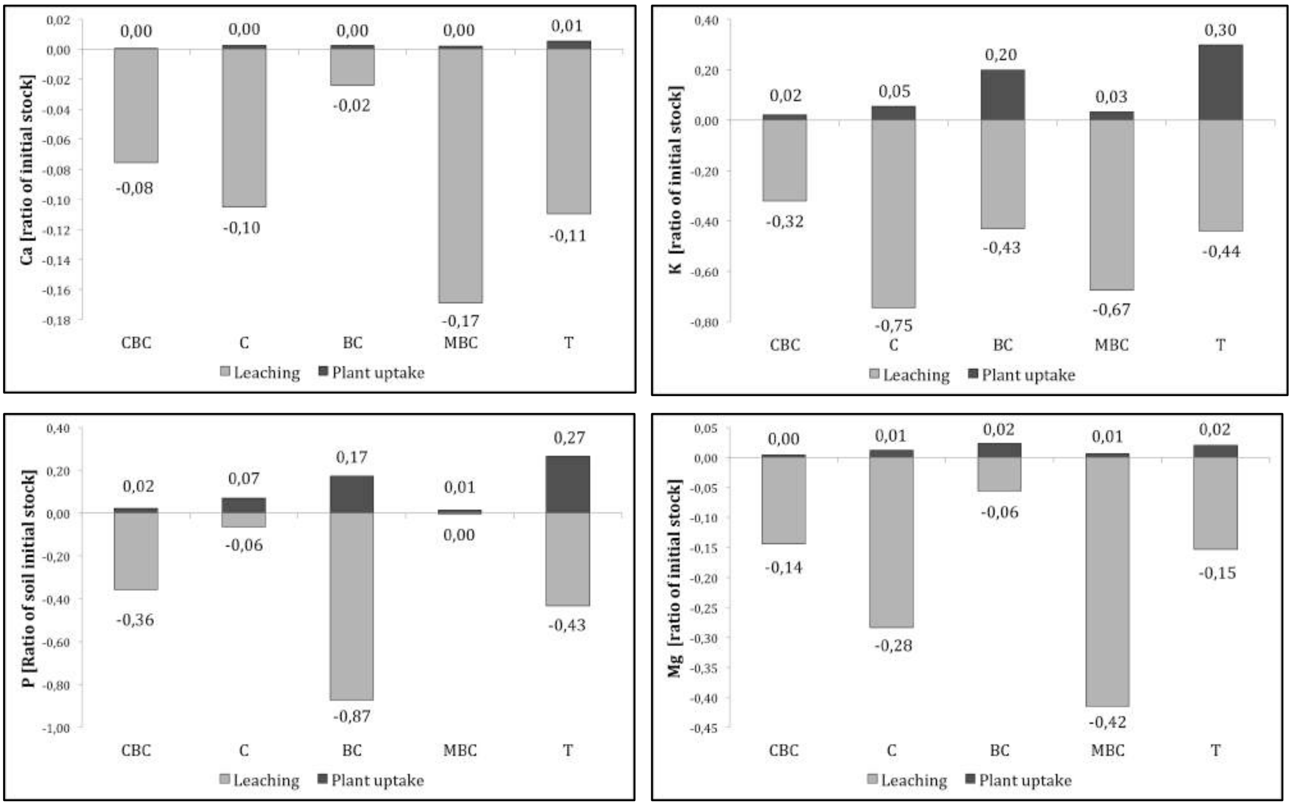


Figure 8: The Ca, K, P and Mg plant uptake to initial stock ratio and total leaching to initial stock ratio (here showed as a negative value) for all the different treatments: co-compost (CBC), compost (C), pristine biochar (BC), mineral loaded biochar (MBC) and the control (T).

4.3.1 Organic biochar activation

The co-compost treatment had different impacts on the nutrients cycle depending on the analysed nutrient (Figure 8). K and Mg retentions were improved as compared to the compost amendment but increased slightly compared to the control. However, Ca distribution did not especially vary between the modalities. Less K and Mg leachings were also noticed with the pristine biochar amendment. Indeed as mentioned previously, the biochar has a high reactive surface area (Brennan *et al.*, 2001) which is able to bind with cations. Moreover, carboxyl functional groups may have increased on the surface of the co-composted biochar after incubation (Cheng, Lehmann and Engelhard, 2008; Khan *et al.*, 2016) but less probably in the biochar pores since the compost temperature reached only an average of 29°C (Cheng *et al.*, 2006). Still, co-compost amendment had not systematically lower leaching to stock ratio than pristine biochar. Thus, an augmentation of the surface reactivity of the biochar cannot be concluded.

However, despite a more efficient nutrients retention of the CBC treatment compared to C and T treatments, ryegrass shoots did not assimilate higher quantities of K and Mg. Although the Mg plant uptake to stock ratio was quite similar for all treatment, the K plant uptake to stock ratio for CBC was of 0.02 while it was of 0.20 and 0.30 for the pristine biochar amendment and the control column respectively (Figure 8). This observation is comforted by the dry shoots weights results that show a higher biomass yield for the pristine biochar amendment than for the compost and co-compost applications. Those results are contrasting with the ones found in the literature. Instead, it was expected to have higher yields for CBC (Kammann *et al.*, 2015) or C (Schulz and Glaser, 2012) treatments because the initial nutrients pool was higher.

Nevertheless both biochar composted and compost had a pH_w of 9.9 that may have locally disturbed the soil (Lehmann and Kuzyakov, 2015). Roots were probably less likely to develop around compost particles, which might explain C and CBC yields and low nutrients uptakes.

In addition, Joseph *et al.* (2018) explored the nutrients retention mechanisms in composted biochar beside the surface adsorption. He found that a gradient is established between the pristine biochar particles and the ions charged water in the compost piles. Cations and anions are thus brought into the biochar pores while an organomineral coating is formed on the biochar surface capturing the nutrients inside the biochar pores. This will later explain a release of nutrients in two phases: a first fast one due to the dissolution of salts on the biochar surface (Silber, Levkovitch and Graber, 2010) and a second slower one due to the release of pore captured nutrients. Clogging and size reduction of the co-composted biochar was also reported by Prost (2013) and Khan (2016).

Under the hypothesis that after calcination, the bioavailable elements behind the organomineral coating were quantified, some of this stock was perhaps not available for the plants because it was detained in the pores.

There were no significant differences between the treatments for the leached phosphorus quantities. Nevertheless, the trends in figure 8 suggested that co-composted amended column lost smaller quantities of P than the biochar modality and the control column but larger quantities than the compost amended columns.

In contrast, the co-composted biochar had the lowest P uptake to initial stock ratio (Figure 8). The low P uptake from the plants is due to the cumulated factors of: i) the high pH disturbing the roots distribution near the compost particles, ii) the organomineral coating making an hypothetical P concentration unavailable for the plants in the biochar pores, and iii) mostly because the soil has large concentrations of Fe, Al oxide and Ca that are able to bind tidily with the P making it unavailable for the plants (Crutchik and Garrido, 2011; Houben *et al.*, 2017).

4.3.2 Mineral loaded biochar

The cations retained by the mineral loaded biochar during maceration were not washed entirely during this experiment. However, the MBC treatment showed lower ability to retain cations than the other treatments in terms of relative stocks probably due to the rapid desorption of highly water-soluble nutrients pool on the biochar surface (Figure 8) (Gwenzi *et al.*, 2018). In addition the MBC showed high K and Mg concentrations (K: 25.9 mg/100g; Mg: 3.58 mg/100g) compared to the altered soil (K: 3.8 mg/100g; Mg: 2.99 mg/100g) and the distilled water, i.e. only H_2O. Thus, the cations might have flown down the concentrations gradient, from the NPK loaded biochar to the soil solution. This hypothesis is valid for all added amendments in this experiment. However the higher the concentrations differences, the higher the nutrients quantities exchanged (Khan *et al.*, 2016), as seen in the case of the MBC treatment.

Although most results from the literature showed the opposite (Steiner, Garcia and Zech, 2009; Gao *et al.*, 2017), the mineral loaded biochar did not show high plants uptakes in terms of initial stocks with respectively Ca, Mg and K ratio of 0.00, 0.01 (Figure 8). This might be related to the rapid leaching of the nutrients from the start of the experiment. Indeed during the first ten days of the experiment the Ca and Mg leachings were the highest while the ryegrass plants were still at an early growth stage and might have not been able to use efficiently the nutrients released by the biochar particles. Yet, the biochar release was still too rapid for the plant uptake and would not have been able to provide cations for the entire season (Angst and Sohi, 2013). Moreover, the mineral loaded biochar did not show aptitudes to retain the K fertiliser addition since most of the 120 mg added passed through the column (Figure 3).

In contrast with cations dynamics, the phosphorus dynamic was most surprising. Although biochar surface carries positive sites as well, its anionic capacity is generally twofold to threefold lower than its CEC (Novak *et al.*, 2009; Silber, Levkovitch and Graber, 2010). Also in the case of the maceration, the anions retention cannot be attributed to a coating development over the biochar pores as exposed above. However, biochar retention of anions such as phosphate or nitrate has been observed several times and might most probably be due to the formation of unusual hydrogen-water bonds between the pores surface of the biochar and the hydrated anions (Conte *et al.*, 2013; Kammann *et al.*, 2015). However, the plants did not assimilate the P applied with the biochar nor the P fertiliser (Figure 8) as shown by a plant uptake to leaching ratio of 0.01 (Figure 8). Most likely the P retained on the biochar was released at a slower rate than the cations (Angst and Sohi, 2013) and may have been immobilised by the Fe and Al oxides or by amorphous precipitates with Ca (Steiner, Garcia and Zech, 2009). Yet, it has to be noticed that this fixation occurs although the pH_w between 6 and 7.5 are most suitable for P bioavailability (Havlin *et al.*, 2014; Weil and Brady, 2017).

4.3.3 NH_4^+ leaching dynamics

The initial NH_4^+ concentrations in the soils and the ryegrass shoots were unknown. Hence, the computation of the ratio was not possible. Yet, the leaching observations gave us an insight of the nutrients dynamics for the 5 soil columns treatments (Figure 5).

Before the fertiliser addition, most of percolated concentrations were similar and steady between the treatments except for the MBC. Indeed MBC was enhanced with DAP fertiliser such (Table 4), and thus may have fixed a large amount of NH_4^+ on the negative functional groups of biochar and through capture into biochar micropores (Brennan *et al.*, 2001; Clough *et al.*, 2013). It has already been used for ammonium water removal (Saleh, Mahmoud and Rashad, 2012). However it is important to note that there was no nitrate monitoring in this experiment. Thus, in this case it is impossible to assert if the N was stocked in the soil, in plants, leached out or volatilized under other nitrogenous forms (NO_3^- or NH_3). However NH_4^+ was steadily released from the MBC columns. Besides, the NH_4^+ added with the fertiliser pulse was stocked in the soil and not leached immediately in all treatments.

5. Conclusion

Co-composted biochar and mineral loaded biochar were suggested as new amendments for fertility and production improvements in altered soils in Burkina Faso. Indeed, biochar is considered as an efficient nutrients carrier, retaining nutrients in the soil and making them available for a longer time period in the soil.

Its abilities to decrease the nutrients leaching and to improve the nutrients bioavailability in soils were tested through soil columns experiments. Biochar maceration with NPK fertiliser increased the biochar nutrients concentrations except for Ca that was probably lost by desorption. Analysis of the co-compost did not show any difference with the compost.

All amendments did increase the Mg and K nutrient status concentrations whereas no change was reported for C_{org} and Ca. Likewise, no enhancement was observed for the soil P concentration except for the NPK mineral loaded biochar amendment.

Nutrients leachings and plants uptakes results contrast with those tendencies of soil fertility improvements. Indeed, biomass yields and nutrients uptakes were not significantly different between the treatments. The co-compost decreased the cations leaching, but only slightly while compared to the control, and did not decrease the P leached quantity. In contrast, the mineral loaded biochar decreased the P leaching but not the cations loss. The pristine biochar application had similar impacts than the co-composted biochar.

Further researches are needed to understand the mechanisms involved in this precise case and to highlight the interactions between the different types of biochar activation, this particular soil and the plants.

6. Perspectives

In order to improve the quality of the results, the precision of some processes could be enhanced. For instance, bioavailable elements were quantified after EDTA extraction which overestimated the directly bioavailable nutrients stocks of the soil and influenced the ratios used in the discussion. To adjust those concentrations quantification of directly bioavailable nutrients could be done with $CaCl_2$ extraction.

To develop our understanding of the mechanisms behind the nutrients adsorption/release in soils amended with enhanced biochar, our results can be compared to kinetic extraction batch experiments. Isotopes could also be used to attest the allocation of the nutrients released by the biochar.

Furthermore, it should be underlined that the compost used in this study was essentially composed of manure, while composts in Burkina Faso are mostly made of lignocellulosic materials and might impact differently the biochar nutrients adsorption and its future characteristics.

This study set the first explanatory steps to understand the effects of nutrients loaded cotton biochar on soils in Burkina Faso. Although the results of this study cannot be directly extended to larger areas, they will be helpful for interpretation of future field study results and comprehend how this new technology could be used effectively to solve soils fertility problems encountered in West Africa.

Part 2: Supplementary information

1. State of the art

1.1 Biochar

Biochar is the solid product of organic materials incomplete combustion with few or no oxygen (i.e. pyrolysis). There is no major difference between regular charcoal and biochar except that this last one is produced explicitly to serve as amendments on fields (Lehmann *et al.*, 2011; Scholz *et al.*, 2014). This technology is inspired from extremely fertile anthropogenic soil formed during the Pre-Columbian era, the Amazonian Dark Earth (ADE). Indeed, the high fertility of ADE compared to the surrounding tropical soils was attributed to the incorporation of stable organic matter in the soil of which included a considerable charcoal concentration (Lehmann *et al.*, 2003; Tenenbaum, 2009).

Biochar has a high concentration of stable carbon (Qayyum *et al.*, 2014) formed mostly of aromatic structures (Kuppusamy, Thavamani and Megharaj, 2016) and have been suggested as a suitable solution for carbon sequestration in soils (Woolf *et al.*, 2010) and for energy production. In addition biochar may improve soils quality and crops productivity (Scholz *et al.*, 2014). Therefore, biochar was often reported as a Win-Win-Win scenario for sustainable energy production, climate change mitigation and food insecurity reduction (Laird, 2008; Sohi *et al.*, 2010).

Biochar is a low density material with a porous matrix which has been reported to increase sandy soils water retention and to lower bulk density in high clay soils allowing better roots penetration, soil aeration and drainage (Laird, 2008; Downie, Crosky and Munroe, 2009). Hence, biochar is beneficial for soils physical properties, as other organic amendments (Chan *et al.*, 2007). Moreover, by increasing water retention, biochar decreases nutrients leaching as attested by Laird (2010).

Furthermore, biochar has a high nutrients adsorptive capacity due to its large number of micropores (diameter < 2nm), and the functional groups covering the biochar surface (Joseph *et al.*, 2010). Higher soil CEC increase have frequently been reported after biochar application (Sohi *et al.*, 2010; Houben *et al.*, 2017). In contrast, Mukherjee *et al.* (2013) reported that the fresh biochar CEC is not always high and range from none to around 70 cmolc kg^{-1}.

However, biochar CEC is expected to increase over time as biochar stays in contact with oxygen and water (i.e. biochar ageing) (Joseph *et al.*, 2010). Alteration of biochar by oxygenation increases the carboxylic and phenolic functional groups and decreases the positive charges on the biochar surface (Cheng *et al.*, 2006). Biochar nutrients adsorption capacity might increase over time. Biochar also has a liming effect on soils due to its alkaline salts and ashes contents.

However the intrinsic properties of biochars mostly depend on its feedstock and pyrolysis temperatures (Lehmann and Joseph, 2009). The initial biomass influences surface area and pH. For instance, manure biochars contain higher nutrients concentrations and have higher pH than woody biochars. In opposition, wood or crop residues biochars have a higher surface area and carbon content (Cantrell *et al.*, 2012). Indeed, the physical structure of the pyrolysed organic matter (OM) is maintained. Capillaries of woody biochars will be maintained and hence, serve as a porous material in comparison to sludge which has less intrinsic structure.

High temperatures biochars (≥ 550°C) are expected to have more stable structures and thus to remain longer in soils. On the contrary, low temperatures biochars (< 550 °C) will develop more interaction with soil components (Steinbeiss, Gleixner and Antonietti, 2009; Joseph *et al.*, 2010).

In addition, feedstock initial nutrients content and pyrolysis temperature also influence biochar nutrients content as volatilisation of nutrients occurs at different temperatures. Carbon (C), nitrogen (N) and sulphur (S) start to volatilise at temperatures below 375 °C and potassium (K) and phosphorus (P) between 700°C and 800°C. Dissipation of calcium (Ca), manganese (Mn) and magnesium (Mg) occur at temperature over 1000°C (Lehmann and Joseph, 2009).

Biochar application influence soils environment and plants roots system, fauna and microbial populations (Lehmann *et al.*, 2011). The abundant biochar pores might provide habitat for soil microorganisms, especially bacteria, actinomycetes and arbuscular mycorrhizal fungi (Thies and Rillig, 2009). Alkalinity increase induced by biochar may contribute to increase microbial abundance; However, in contrast, such augmentation might be unbeneficial for fungi abundance (Lehmann *et al.*, 2011). Also microbial activity might be negatively impacted by biochar application depending on soil types. For instance, Ameloot *et al.* (2015) observed an augmentation of microbial activity in soils with high OM content compared to soils with low OM content. Furthermore, this study showed that microbial response to biochar depended on the microbial abundance and communities.

As stated previously, biochar properties can vary widely. However, soils types and environmental conditions influence widely the biochar effects. For example, biochar increases crop yields on acidic soils with low nutrient supply, like tropical soils, in comparison to biomass grown on temperate soil with more neutral pH and high nutrient supply (Jeffery *et al.*, 2017). Similarly, Gul *et al.* (2016) reviewed that high CEC biochar apply in a nutrient-rich soil would decrease the nutrients leaching while the same biochar amended in nutrient-poor soil could significantly decrease the bioavailability of nutrient for crops.

1.2. Biochar activation

Few studies have combined biochar with nutrient-rich materials in order to offset the initial low CEC and nutrients content of biochar (Prost *et al.*, 2013; Kim, Hensley and Labbé, 2014). Although fresh made biochar has low nutrients concentration, it can be a proper ions carrier by making them available in the soil for longer time period (Khan *et al.*, 2008; Manikandan and Subramanian, 2015; Schmidt *et al.*, 2017). This concept of boosting biochar nutrients concentration has been wearing different names in the literature such as “enhanced biochar” (Schmidt *et al.*, 2017), “nutrient loaded biochar” (Joseph *et al.*, 2018) and sometimes as “activated biochar” when associated with composting (Bonanomi *et al.*, 2017).

1.2.1 Mineral loading of biochar through maceration

Combining biochar with fertiliser can be done through maceration of biochar in NPK fertiliser solution (Manikandan, 2013; Gwenzi *et al.*, 2018). For example, Manikandan *et al.* (2015) mixed different kinds of nitrogenous fertilisers and more specifically urea in equal concentration with biochar. He found that urea loaded biochar retained nitrogen longer than in separated applications of biochar and urea. Likewise, Khan (2008) boosted oak wood biochar with NPK fertiliser and observed a slow and steady nutrients release from mineral loaded biochar under continuous leaching. Furthermore, Schmidt (2017) reported yields improvement up to 20% with an application of NPK enhanced biochar compared to standard NPK fertilisations.

However, few studies concerning the adsorbing/releasing mechanisms occurring during maceration and in soils have been conducted so far.

The bonds between biochar and nutrients in solution can be of different types: i) water bridges between biochar oxygenated functional groups and the hydrated surface of ions (Conte *et al*., 2013; Kammann *et al*., 2015), ii) ionic bonds with positives or negatives sites on the biochar and iii) covalent bounds (Schmidt *et al*., 2017). It has also been reported that ions could remain in biochar by water capillarity retention. Then, the slow release depends on pores size and networks (Khan *et al*., 2008).

1.2.2 Organic activation through co-composting

a) Composting

Bernal *et al*. (2009) defined composting as "a biooxydative process involving the mineralisation and partial humification of the organic matter, leading to a stabilised final product, free of phytotoxicity and pathogens and with certain humic properties". Multiple types and processes of composting exist (Fischer and Glaser, 2013), however, composting can be considered as a discontinuous process that goes through 4 different temperature stages (Insam and de Bertoldi, 2007) : the mesophilic stage, the thermophilic stage, the cooling stage and the maturation stage.

During the mesophilic stage, primary decomposers break down easily degradable compounds. Their aerobic metabolism induces temperature rising from 25°C to 40°C. Thermophilic bacteria and actinomycete decompose labile carbon over 40°C. Passing 65°C most of bacteria and fungi are destroyed and the temperature may still increase (>80°C). During the thermophilic stage, human and plant pathogens, weed seeds and insect larva are killed. The cooling stage is characterised by a decrease of temperature. A second generation of mesophilic organisms colonise the compost. This time bacteria and fungi decompose essentially starch and cellulose. Then, the compost temperature keeps decreasing and stabilises below 25°C and the maturation stage starts. Concentrations of humus-lignin complex compounds increase as the fungi fraction and macro flora and fauna colonise the compost (Insam and de Bertoldi, 2007; Fischer and Glaser, 2013).

Once the compost has reach the maturity stage it is important to assess its stability and maturity and to make sure the product has the adequate quality to be applied as a fertiliser. Several parameters and criteria, either biological, physical or chemical, have been suggested to attest the compost maturity, associated with the phytotoxicity, and stability, linked with the microbial compost activity (Le Page *et al*., 2007). However none of the proposed methods in the literature could be applied to every case given the variety of feedstock and processes used for composting.

b) Biochar co-composting

Synergism has been reported between compost and biochar while biochar is incorporated during the composting process (Fischer and Glaser, 2013). On the one hand, biochar is a suitable bulking agent for the compost, allowing aeration, water retention and maintain of the structure of the compost pile (Bernal, Alburquerque and Moral, 2009; Steiner *et al.*, 2011). Biochar incorporation in the compost pile allows the reduction of nitrate (NO_3^-) other nutrients leachings (Steiner *et al.*, 2010) and the maintain of the compost humidity. Some studies also revealed that biochar in the compost helped reducing the ammoniac volatilisation (Steiner *et al.*, 2010; Clough *et al.*, 2013).

On the other hand, the temperature increase during the thermophilic stage speeds up biochar ageing, increasing the number of functional groups and hence the biochar CEC (Khan *et al.*, 2016). Furthermore, adding biochar to composting boosts biochar nutrients concentrations before its application in soils. The previous bonds mechanisms described for the mineral loading of the biochar are also valid in the case of co-composted biochar. In addition, studies found out that interactions between compost OM and biochar might also be responsible for nutrients retention (Figure 9). Indeed, during the composting process, concentrations gradients are established between the nutrient-poor solid material (i.e. biochar) and the compost solution. Moreover, organomineral coatings are formed on the surface of the biochar clogging the pores and trapping the nutrients inside the biochar pores (Prost *et al.*, 2013; Schmidt *et al.*, 2015; Joseph *et al.*, 2018). This new organomineral complex might be the site of new chemical bonds increasing the adsorption potential of the biochar particle (Conte and Laudicina, 2017; Schmidt *et al.*, 2017).

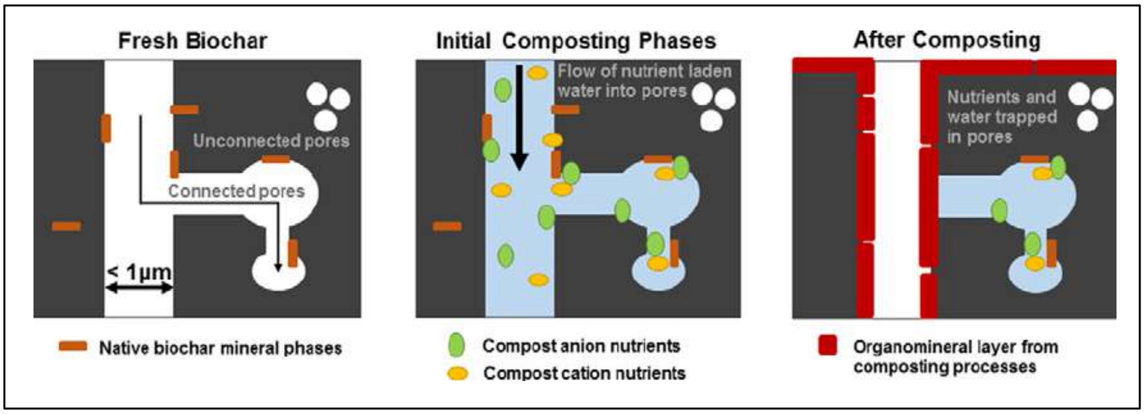


Figure 9: Conceptual model of the organic coating formed on the biochar pores during the composting process. Retrieved from Joseph *et al.* (2018)

1.3 Nutrient cycles and biochar impact

1.3.1 Nitrogen Cycle

Nitrogen (N) is mostly found in the atmosphere under N_2 form. The bonds between those 2 atoms are strongs making this gas unavailable for most life beings. However, different processes modify N to make it available for plants. The nitrogen cycle (Figure 10) is composed of a lot of transformations and translocations but can nevertheless be summed up in 8 phases: mineralisation, nitrification, immobilisation, fixation, volatilisation, leaching, uptake and denitrification (Havlin *et al.*, 2014).

Soils main sources of nitrogen are plants and animal residues, electrical discharge from the atmosphere, legumes roots nodule fixation, and industrial fertiliser. Microorganism break down large organic molecules and the N is mineralised into ammonium (NH_4^+). Much of the NH_4^+ produced is transformed into nitrate (NO_3^-) under the action of nitrifying bacteria during nitrification. This step results in the transformation of NH_4^+ in nitrite (NO_2^-) and successively in NO_3^-. The reverse reaction, immobilisation, occurs when NH_4^+ and NO_3^- are transformed into organic N forms. NH_4^+ and NO_3^- are the forms of mineral N that can be absorbed by plants.

NH_4^+ might be stocked into soils by fixation on clay 2:1 materials but it can also be volatilised as NH_3 in the atmosphere. Volatilisation of NH_3 is more likely to occur when soils pH and temperatures are respectively over 7 and 16°C (Weil and Brady, 2017). Nitrogen might also be lost from the soil by leaching of NH_4^+ and NO_3^- or by denitrification of NO_3^- into nitrogenous gases (N_2, NO_2, NO) by the action of denitrifying bacteria (Havlin *et al.*, 2014; Weil and Brady, 2017).

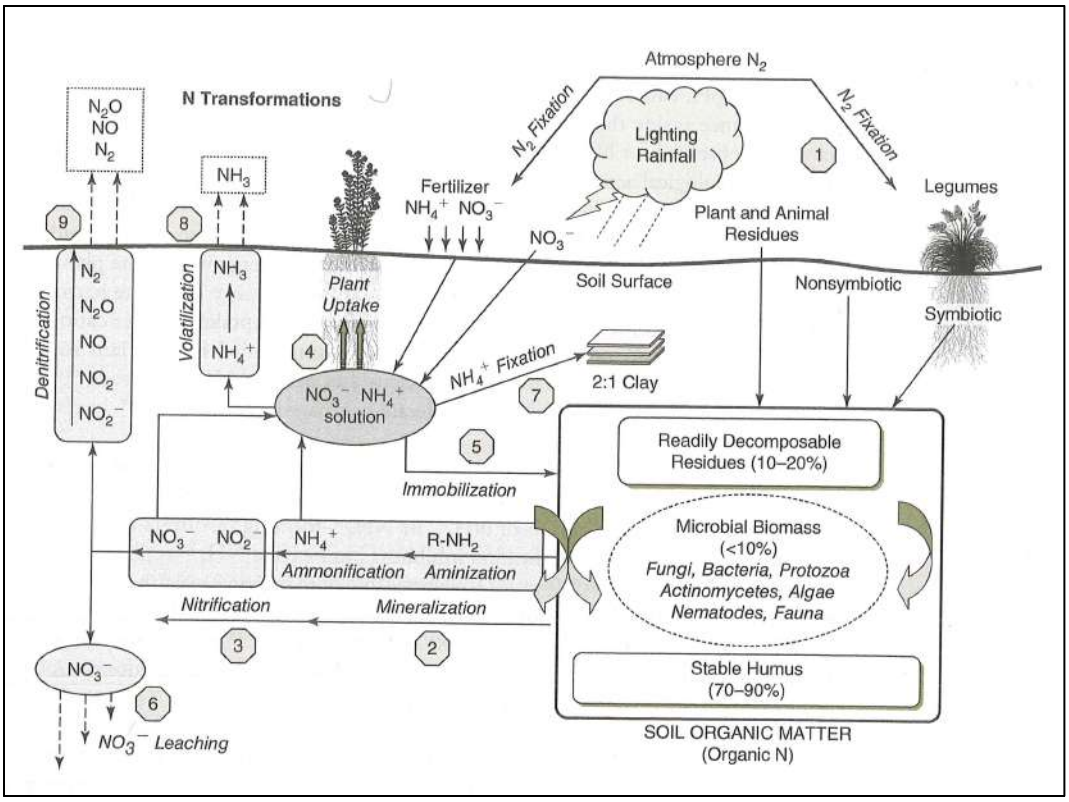


Figure 10: Nitrogen cycle retrieved from Havlin *et al.* (2014)

1.3.2 Biochar impact on the nitrogen cycle

Biochar has been reported to impact the nitrogen cycle in different ways. Biochar amendments increase the C stocked in the soil increasing the C/N ratio. Hence, it reduces the N mineralisation rate and its bioavailability (Jones and Parsons, 1970; Lehmann *et al.*, 2003). In opposition, Ameloot *et al.* (2015) found an increase of N mineralisation when manure biochar was applied in soil although assimilation rate was increased when pine wood biochar was amended. Then, N mineralisation might depend on the biochar feedstock as well as the biochar properties developed above (cf. -Part 2, 1.1 Biochar). Biochar retains NO_3^- and NH_4^+ through water retention and ions adsorption and hence prevents the N losses by leaching. NH_4^+ and NH_3 fixation on biochar functional groups allows reducing NH_3 volatilisation (Spokas, Novak and Venterea, 2012; Clough *et al.*, 2013). Nevertheless the liming property of biochar might benefit the formation of NH_3 rather than NH_4^+ fixation after fertilisers application (Schomberg *et al.*, 2012). The nitrification can be decreased by an application of biochar as shown by Taghizadeh-Tossi *et al.* (2012). The nitrification rate decreased as a function of the increasing application rate of biochar, probably because biochar reduces the availability of NH_4^+ for the ammonia oxidisers (Taghizadeh-Toosi *et al.*, 2012).

In addition biochar may have a negative impact on denitrification and thus might decrease nitrogenous greenhouse gases emissions. Martin *et al.* (2014) observed a 42% decreased of the N_2O emission on an amended field after an application of wood biochar (3%) in a sandy loam. In contrast, Sheer *et al.* (2011) observed no impact of a biochar application on a Australian Ferralsol pasture to reduce green house gases reduction.

1.3.3 Phosphorus cycle

Phosphorous is also an element essentials for all living beings. However, bio-available P is often one of the growth limiting factors in the soil (Weil and Brady, 2017). The phosphorus cycle is shown on Figure 11.

The main sources of P in soils come from primary mineral weathering, industrial fertilisers and residues decomposition. Animals and plants residues are broken down by the microorganisms and transformed in ortho-P (i.e. HPO_4^{2-} and $H_2PO_4^-$) during the process of mineralisation. Ortho-P are the only bioavailable P forms and yet, they are quite immobile in the soil (Weil and Brady, 2017).

P reacts strongly with other elements in soils such as Fe^{2+}, Al^{3+} and Ca^{2+} but also with the Fe and Al oxy-hydroxide. Those reactions depend of the soil pH, in acidic soils, precipitation occurs with Fe and Al, whilst in more alkaline ($pH > 7.5$) soils P precipitate with Ca. P can also be adsorbed on clay 1:1 such as kaolinite (Havlin *et al.*, 2014). However, different degrees of P fixation have to be considered. Indeed, P can be weakly adsorbed by anion exchange reaction on the clay or oxide surface forming an outer-sphere complex. Then, P is still bioavailable for the plants. Yet, when this P ion replaces one or two hydroxyls on the oxide or clay surface, it becomes an inner sphere complex and is not available for plants anymore (Weil and Brady, 2017).

Phosphorus is more available with pH value ranging from 6 to 7 and is thus, more leachable (Weil and Brady, 2017).

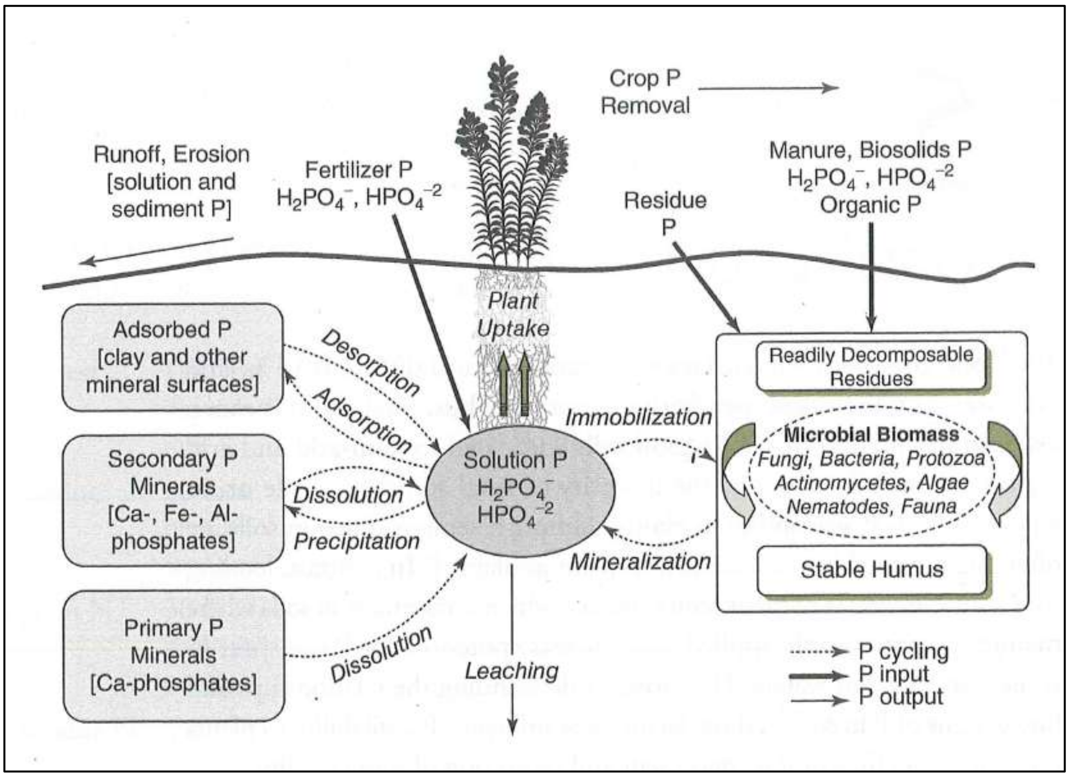


Figure 11: Phosphorus cycle retrieved from Havlin *et al.* (2014)

1.3.4 Biochar impact on the phosphorus cycle

Biochar produced at temperatures below 700°C might still contain P concentrations similar to their feedstock since P only starts volatilising at that temperature (Atkinson, Fitzgerald and Hipps, 2010). Hence Biochar might provide a direct source of P to the plants.

Furthermore, biochar can indirectly modify the P bioavailability. Indeed biochar liming capacity increases the pH of acidic soil. Then, the soil P fixation capacity is decreased due to the dissolution of the Fe and Al oxide (Houben *et al*., 2017; Weil and Brady, 2017). Also at higher pH, organic ions are competing to bind with Ca, Al and Fe resulting in a decrease of P fixation (Houben *et al*., 2017).

In opposition, applying biochar to a neutral or alkaline soil might cause over-liming and the immobilisation of P anions by precipitation with Ca (Jeffery *et al*., 2017).

Biochar might decrease the P leaching by water bridges with phosphates (Conte *et al*., 2013; Kammann *et al*., 2015).

1.3.5 Potassium cycle

Potassium (K) remains under ionic form in the plants and plays a regulatory role in enzymes activation and stomata opening during photosynthesis (Van Brunt *et al.*, 1998). The optimum K concentration of plants growth is between 1 to 10 ppm.

As displayed on Figure 12, the K inputs in the soil come from the microbial recycling of residues and fertiliser application. Micas and vermiculite (1:2 clays) secondary mineral deriving from micas and feldspar (primary minerals) weathering are also a source of K^+ in soils. The cations can be fixed in clays interlayer or on the clay surface. They are available on both locations to rebalance the soil solution and K^+ are released by diffusion and cations exchange capacity (Havlin *et al.*, 2014).

In contrast with ortho-P anions, K^+ cations are mobile elements subject to leaching. In particular under acidic pH, K leaching is exacerbated because K^+ is less likely to compete with Al^{3+} for adsorption on the clay complex (Weil and Brady, 2017). K^+ leaching is also more important in sandy soils with mostly kaolinite clay (1:1 clay) that does not adsorb K^+ in the interlayer.

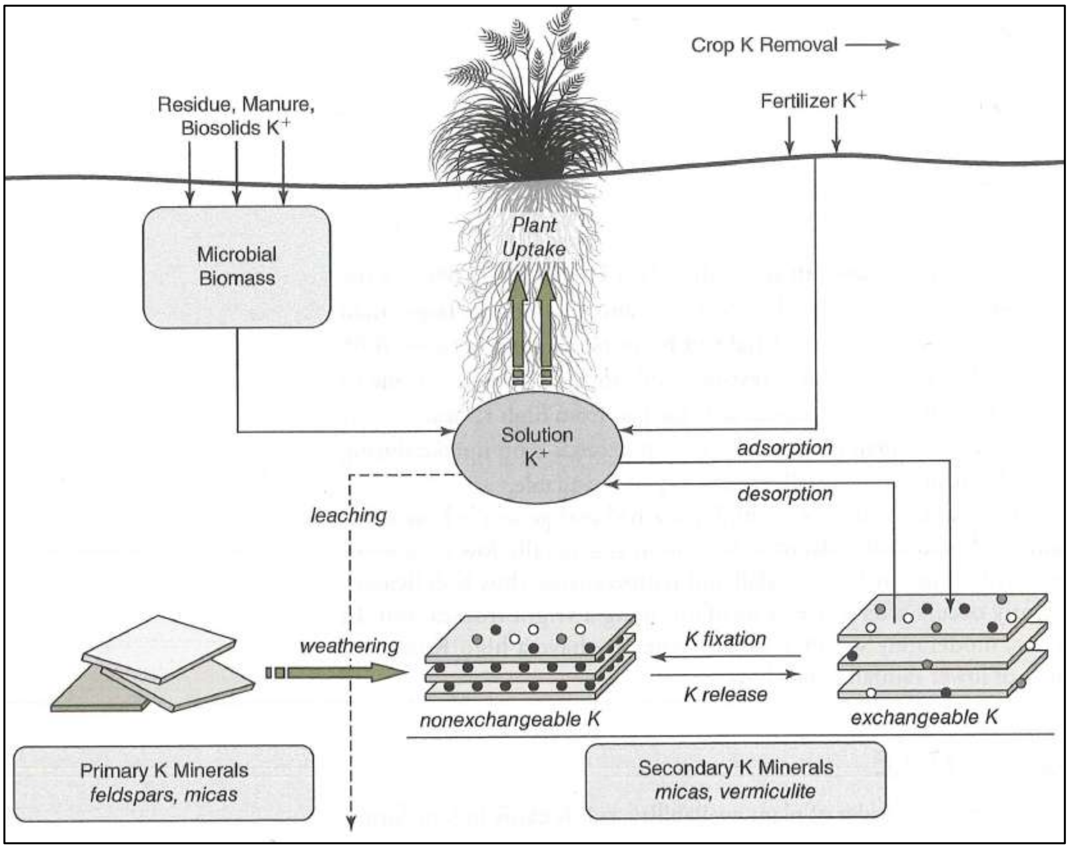


Figure 12: Potassium cycle retrieved from Havlin *et al.* (2014)

1.3.6 Calcium and Magnesium cycle

Ca and Mg are both important elements for plants. Ca is essential in the cell wall structure of the plants. Mg is also present in plants tissues and plays a role in proteins synthesis. The Ca and Mg cycles are shown on Figure 13.

Different pools are supplying Mg and K to the plants. Similarly to the above mentioned nutrients cycles, the source of Mg and Ca are industrial fertilisers, liming materials, microbial broken down residues and weathering of primary materials. Ca^{2+} cations are released from weathering of anothrite, pyroxene, amphibole and calcite while Mg comes from of dolomite, horneblende, biotite, olivine and serpentine. Clays such as vermiculite also have a high Mg content and can be a source of Mg. The Mg and Ca cycles are a lot similar to the K cycle. Those cations are adsorbed or desorbed on the clays depending on the Ca and Mg equilibrium of the soil solution or precipitate as secondary minerals. Ca^{2+} and Mg^{2+} are highly mobile and are subject to leaching (Havlin *et al.*, 2014; Weil and Brady, 2017).

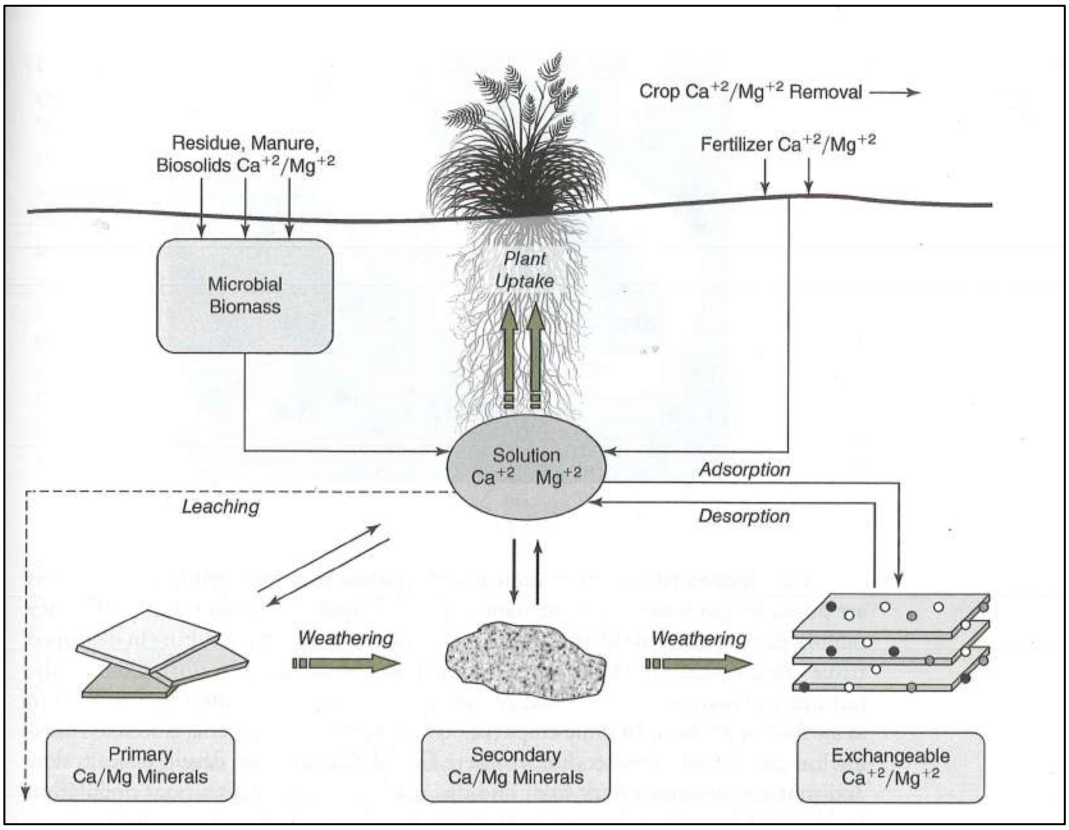


Figure 13: Calcium and magnesium cycle retrieved from Havlin *et al.* (2014)

1.3.7 Biochar impact on the K, Ca and Mg cycles

While some studies have been carried out on the impact of biochar on the N and P cycling, very few focused on the K, Ca and Mg cycles.

Biochar may increase the retention of cations such as K, Ca and Mg by increasing soils CEC (Biederman and Harpole, 2012; Major *et al.*, 2012) or by modifying water fluxes in soils (Laird *et al.*, 2010). Nevertheless, higher leaching rates of K have been noticed as a function of biochar application rates, most probably because of the usually high concentrations of K in the biochar (Lehmann *et al.*, 2003). Kinetic batch experiments highlighted a fast solubility of the water soluble K salts fixed on biochar surfaces (Angst and Sohi, 2013). Also biochar liming capacity can improve the availability of nutrients in acidic soils. Plants assimilated higher K^+ and Ca^{2+} quantities in response to biochar application (Biederman and Harpole, 2012).

2. Materiel, methods and results: additional information

2.1 Compost monitoring

2.1.1 Compost setup

The materials (straws, old manure and fresh manure) came from the experimental farm of Gembloux Agro-Bio Tech. The biochar came from Bobo-Dioulasso, Burkina Faso as explained in the above section (Figure 14).

As seen above, the composting technique was inspired from the Berkley rapid composting method (cf- Part 1, 2. Material and Method). However, the request of a 1m^3 volume was not respected because of the small amounts of available biochar. To avoid heat loss and preserve the thermic inertia of the compost pile, the compost bins were insulated using insulation sheets (Comfortex®) on the container wall bioreactors like in Das *et al.* (2001) and Wang *et al.* (2016) studies (Figure 15).

A special attention was given in order to mix homogeneously the upper part and lower part of the compost with a shovel during every composts turn over. During that process, the water content of the compost was also tested and if so needed water was added. The tap water used was rested over night to assure chlorine evaporation (www.swde.be). Between the mixing phases, the compost bins were closed with a lead and the compost air contact surface was covered with an insulation sheet.

Figure 14: Proportion of feedstock used for composting. Left: co-compost feedstock; Right: compost feedstock

Figure 15: left: set up of the compost bins; right: insulated inside wall of a bin.

2.1.2 Compost temperature

As seen previously (cf-7.1 State of the art), the composting process can be defined by a rapid rise of the temperature inside the compost pile. In this case no temperature increase was observed (Figure 16). The compost temperature oscillated around an average of 29°C.

A technical problem occurred with the temperature sensor of the compost without biochar from 03/26/18 to 03/28/18. Hence, no data is available for this time period

Even if the absence of temperature increase will affect the biochar organic activation, we can attest the fact that both composts followed the same temperatures dynamic and would undergo the same physical transformation. Most likely, the small composts sizes caused a rapid dissipation of the heat.

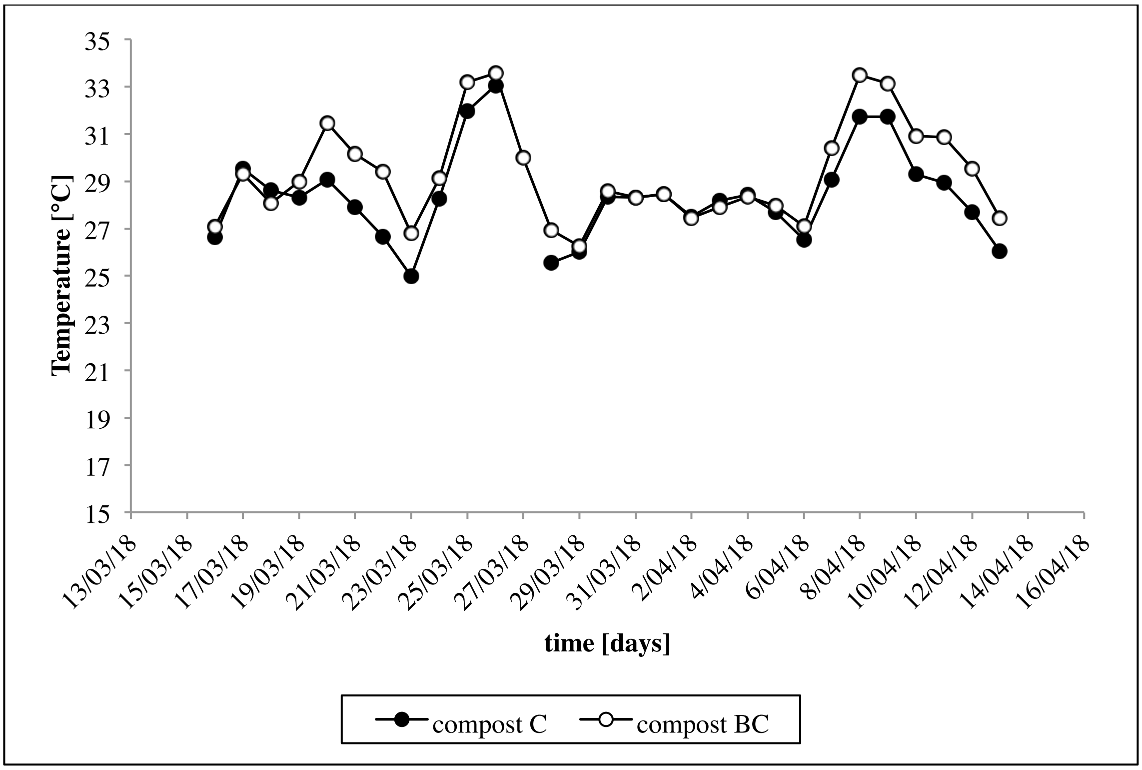


Figure 16: Temperature evolution of compost (C) and the co-compost (BC) over the time of the experiment.

2.1.3 Compost evolution

Figure 17 shows the evolution of the composts over time. Even if straws breakdowns have not been observed, changes in colour and texture occurred.

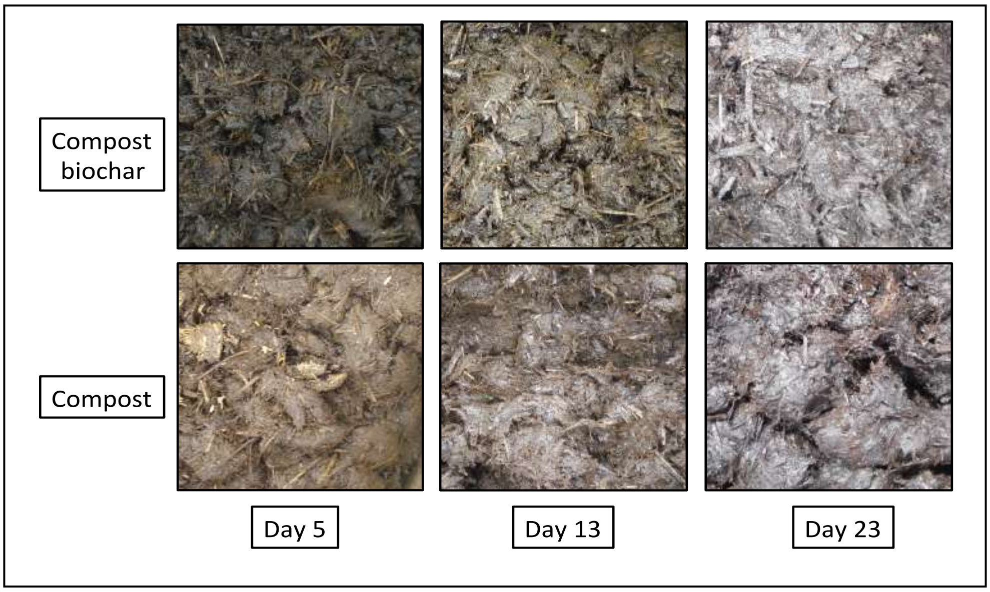


Figure 17 : Co-compost (CBC) and compost (C) colour and texture evolution from day 5 to day 23.

2.2 Bias control

2.2.1 Germination test

The germination rate of the seeds used was studied trough a germination test (Figure 18).

Thirty seeds of *Lolium perenne* L. were placed in petri dishes on humid geotextile and stored out of light for ten days. Then, the germinated seeds were counted and the germination rate was computed as below:

$$Germination\ rate\ = \frac{number\ of\ germinated\ seeds}{number\ of\ total\ seeds}$$

The average germination rate of the ryegrass seeds was of 48% ± 20% (n=10) showing an important variability.

Figure 18: Germination test setup, each 10 cm petri dish contained 30 seeds on humid geotextile, n=10.

2.2.2 Bias from the filtration layer

An inconsistency was noticed while measuring the pH of the collected solutions. On average, the pH was at 8.5 ± 0.2 without any distinction between treatments while the initial soil pH values ranged from 6.5 to 7.3 depending on the amendment type. After investigation, it appeared that the glue (Pattex®) used to seal the column stopper were responsible for the alkalinisation of the solutions. To make sure no significant amounts of elements were released in the soil leachate, 3 new columns were made with the draining layer only. The columns were watered every three days as it was done for the other experimental columns and during 18 days. In order to simulate the same time of contact between the draining layer and the distilled water than in the soil column, a plastic tap plugged the water output. It was placed before the waterings and removed 24 hours later. The 1st, 2nd and 6th percolates were analysed for pH values and nutrients concentrations following the same procedures than for the soil leachates (cf – 2.5. Chemical analyses).

The pH of the soils at 5 cm and 15 cm depths were measured, as explained previously, to ensure that the alkaline glues did not dramatically influence the soil solutions. Indeed, an increase of the pH of the soil solutions would have disturbed the nutrients bioavailability. However, the soil pH values, measured at both depths, were approximately the same as initially (Figure 19). Furthermore, a slight tendency for acidification was noticed and might be due to the repeated leaching and the plants acidification for nutrients assimilation. Hence, no influence of the alkaline filtration layer was observed in the soil layers and did not impact the soil nutrients dynamics.

Additionally to solution alkalinisation, the nutrients quantification revealed that the filtration layer released Ca, Mg and K (Figure 20, Figure 22 and Figure 24). To counter this nutrient release, identical for all columns, the measured concentrations were subtracted from the concentrations for all columns (Figure 21, Figure 23 and Figure 25). Thus, the Figure 1, figure 2 and Figure 3 showed in the previous section display the concentrations without bias.

Mg and K quantities were released at slightly decreasing rates and were considered steady to correct the filtration layer bias. The released Ca amounts increased over time. In order to take into account this increase, the bias concentrations over time were computed based on the equation of the linear regression line displayed on Figure 20.

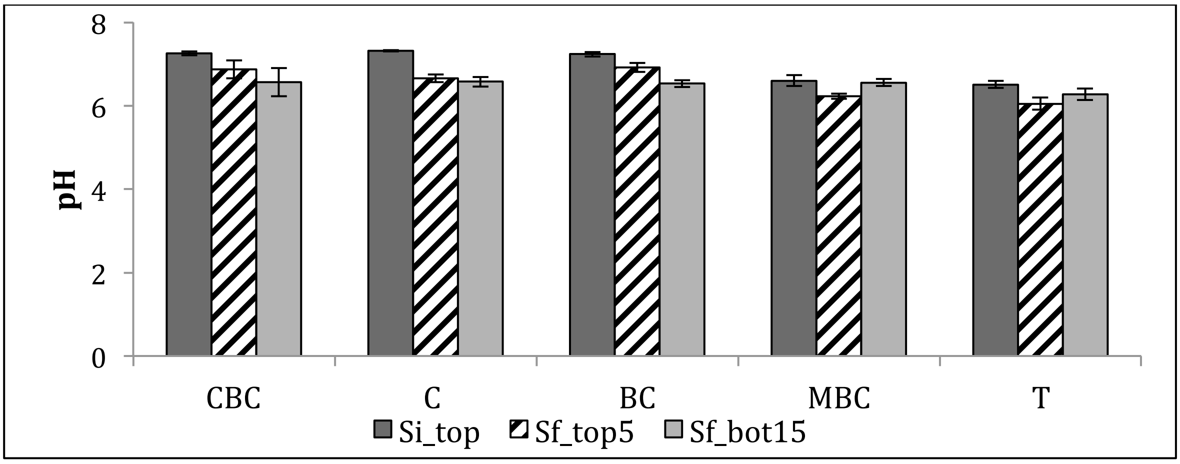


Figure 19: pH$_w$ values of the soil in the top 10 cm of the columns before the experiment (Si_top), and of the soil contained in the columns after the experiment at 2 different depths : 5 cm (Sf_top5) and 15 cm (Sf_bot15). This was measured for all 5 treatments: the co-composted amended soil (CBC), compost one (C), the pristine biochar one (BC), the mineral loaded (MBC) and de control column (T).

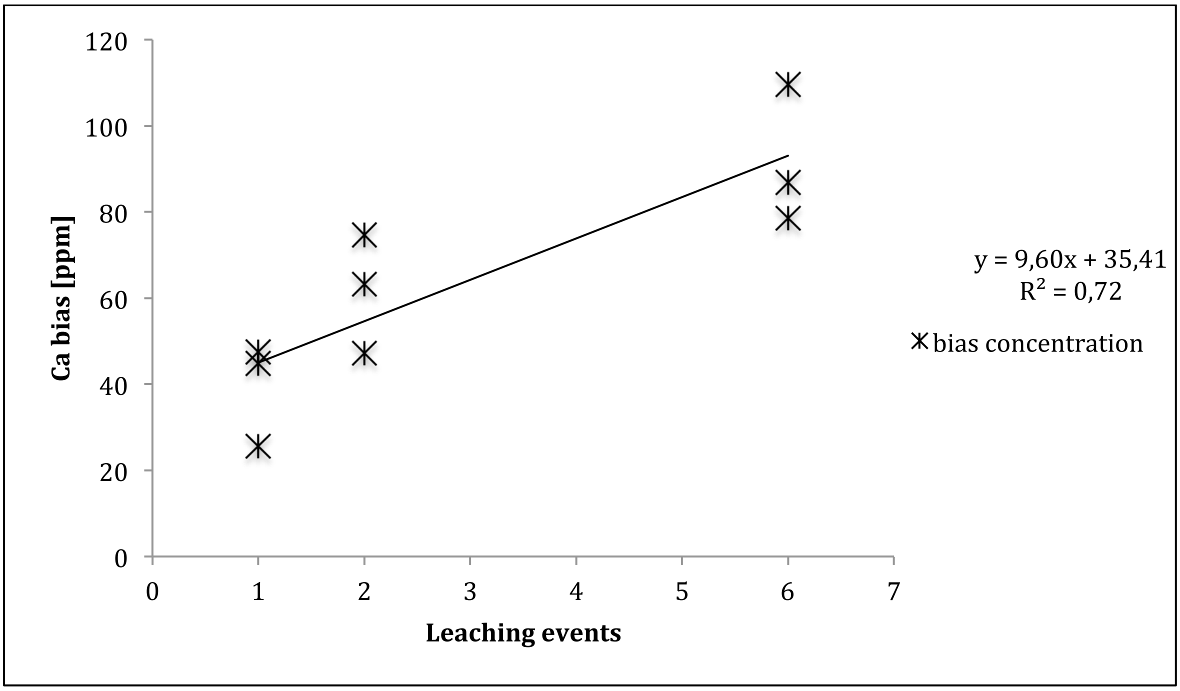


Figure 20: Ca bias concentration for the 1st, 2nd and 6th leaching of the filtration layer.

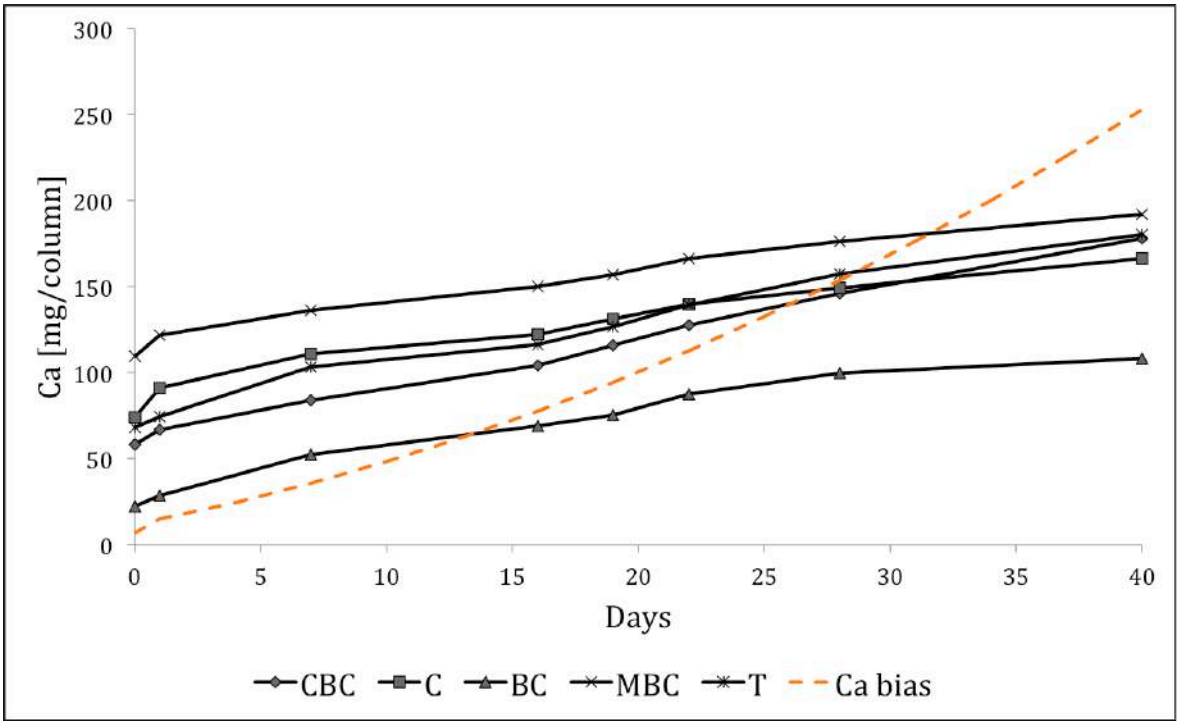


Figure 21: Cumulated Ca leached quantities over time without subtracting the bias for the 5 studied modalities: the co-composted amendment (CBC), compost (C), the pristine biochar (BC) the mineral loaded (MBC) and the control column (T).The orange line represents the cumulated bias over time.

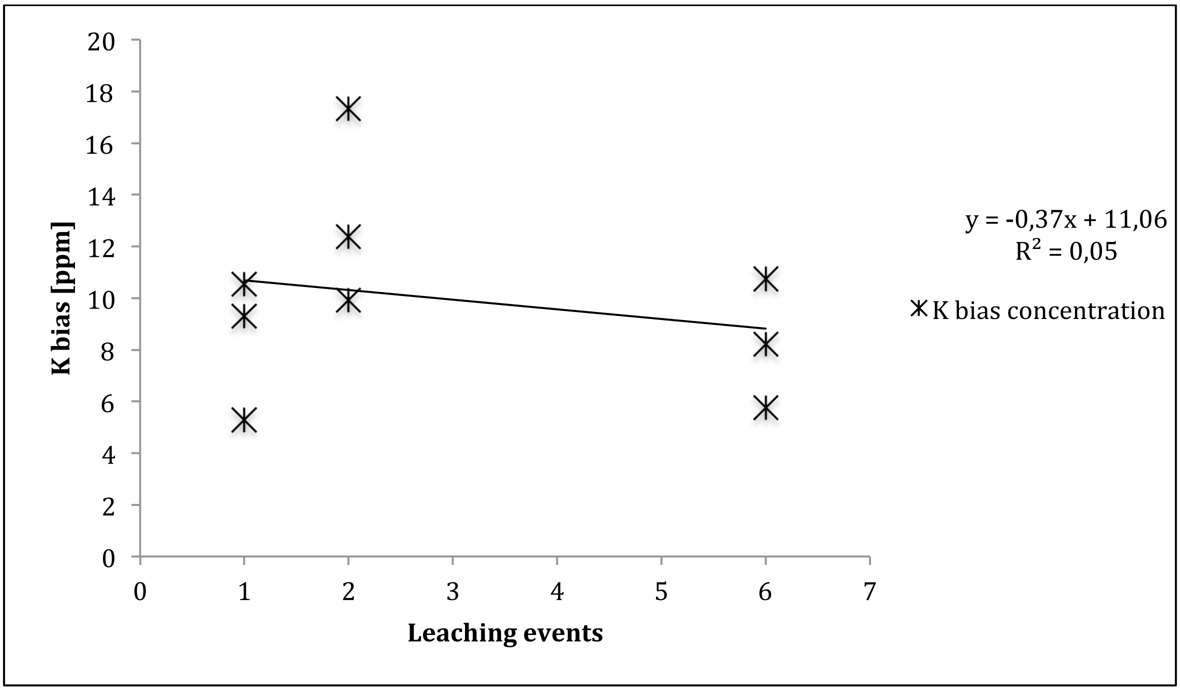


Figure 22: K bias concentration for the 1st, 2nd and 6th leaching of filtration layer

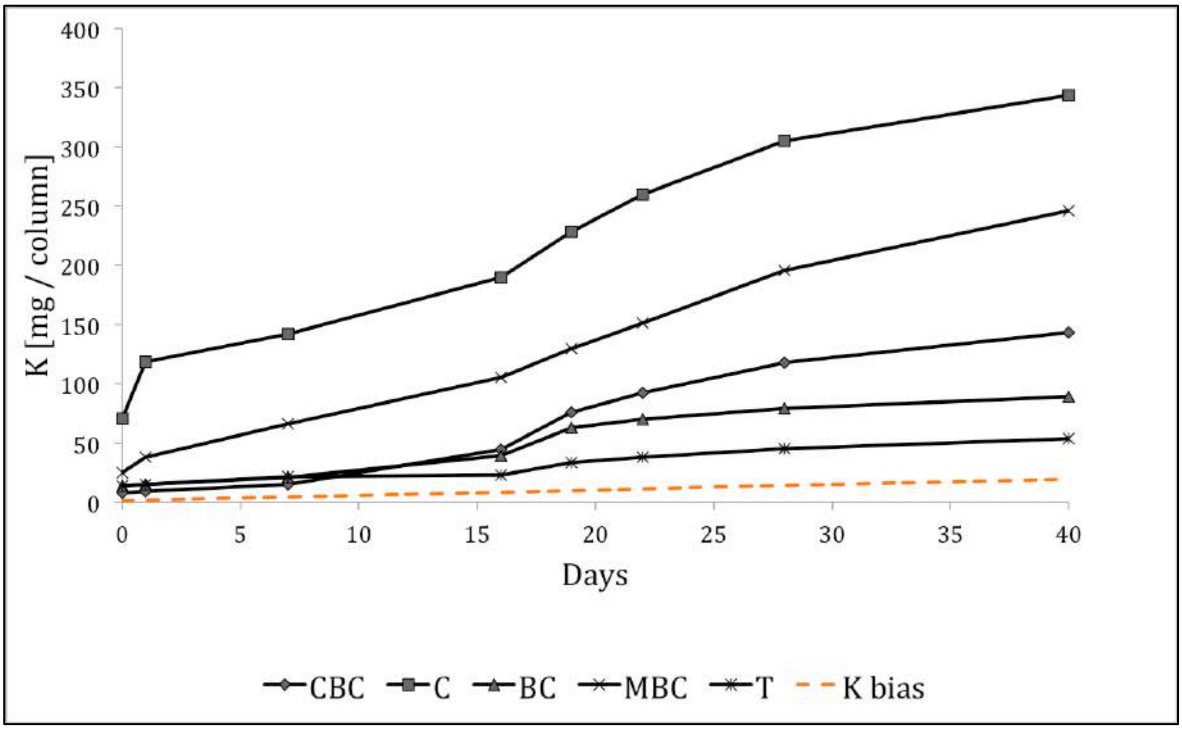


Figure 23: Cumulated K leached quantities over time without subtracting the bias for the 5 studied modalities: the co-composted amendment (CBC), compost (C), the pristine biochar (BC) the mineral loaded (MBC) and de control column (T). The orange line represents the cumulated bias over time.

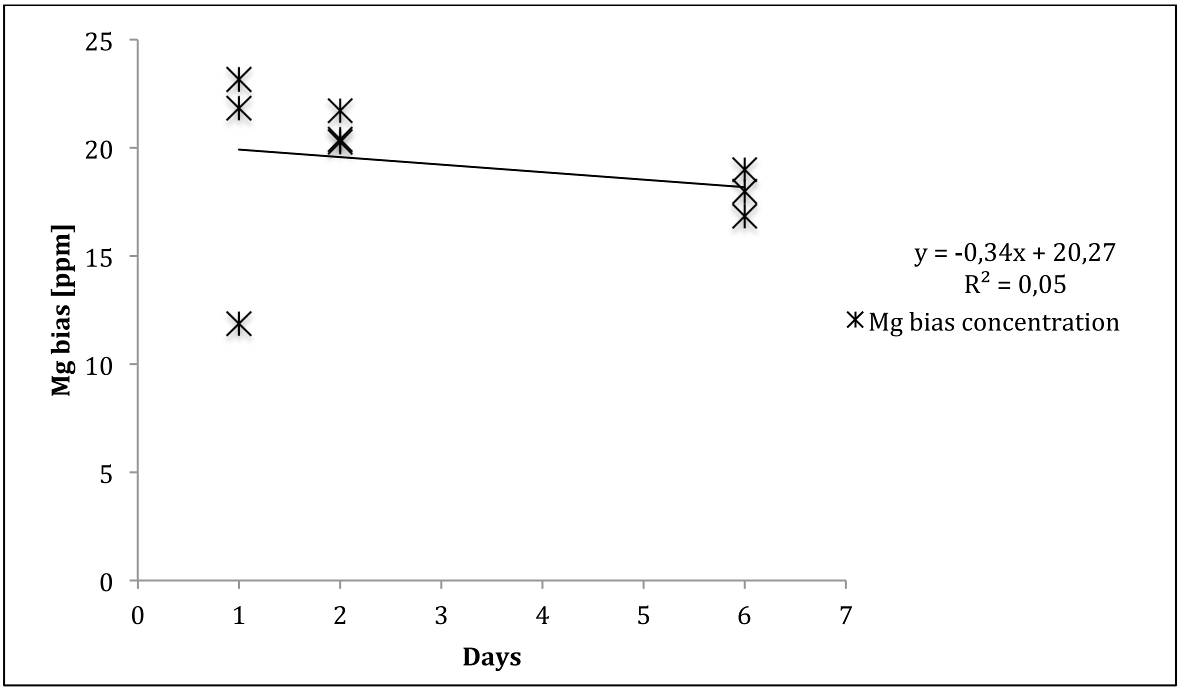


Figure 24: Mg bias concentration for the 1st, 2nd and 6th leaching of filtration layer.

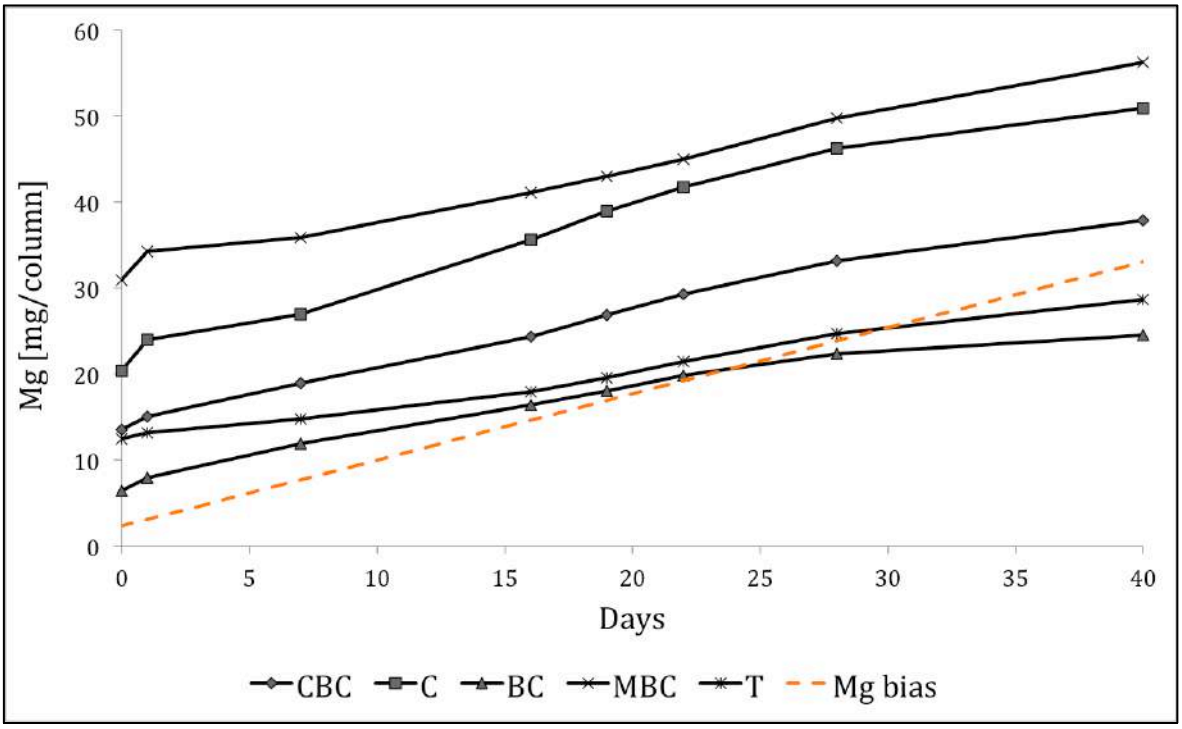


Figure 25: Cumulated Mg leached quantities over time without subtracting the bias for the 5 studied modalities the co-composted amendment (CBC), compost (C), the pristine biochar (BC), the mineral loaded (MBC) and the control column (T). The orange line represents the cumulated bias over time.

2.3 Ammonium quantification

The ammonium concentration of the soils solutions were measured according to the Kjehldal method (KM) as done at the BIOSE department laboratories.

The KM is used to quantify the N_{tot} in soil samples and is composed of two distinct steps. Firstly, the digestion of the soil samples to convert all nitrogenous forms in NH_4^+. Secondly, after steam distillation, NH_4^+ is quantified in the digest through direct titration with hydrochloric acid (HCl) and Tashiro indicator (mixed at equal volumes of methyl red and methyl blue indicators).

The final total N quantity is obtained according the below equation:

$$N_{tot} = v * t * 14 * 100$$

Where: N_{tot} = Nitrogen total of the sample [g]; v = HCl volume used for the titration [ml]; t = molarity of the solution [mol/mL]; 14 is the atomic mass of N.

In this work, the NH_4^+ concentration in the soil solution was quantified by steam distillation with a Büchi® distillation unit. The procedure consisted of a titration of NH_4^+, with HCl (0,01M), collected in a solution of boric acid (H_3BO_3 - 4%-25 ml) after transformation of ammonium ions into ammonia by alkalinisation with sodium hydroxide (NaOH - 35%- 50 ml). Keeney and Nelson (1982) and Van Ranst *et al.* (1999) also used steam distillation to quantify NH_4^+ in soils samples but with different indicators and with an extraction in potassium chloride (KCl).

To attest the reliability of this method, trials were done with a standard ammonium chloride (NH_4Cl) solution and at two different concentrations.

The concentrations of the first solution were established based on the HCl 1M needed for their titration according to:

$$v = \frac{m_{NH4}}{t * M_{NH4}}$$

Where: m_{NH4} = theoretical mass of NH_4^+ in the sample [g] and M_{NH4} = Atomic mass of ammonium [g/mol].

Table 8 shows the theoretical parameters and the results of this titration (Figure 26).

However, the concentrations of the solution were expected to be lower than the ones that could be determined with HCl 1M. Similar trials were done with concentrations determined for titrations with 3.00 ml, 5.00 ml and 8.01 ml of HCl 10 mM (Table 9 and Figure 27).

The results obtained for both ammonium concentrations showed an accurate approximation of the theoretical concentration in the solution and therefore, appeared to be precise enough to be used for ammonium quantification in this study.

Table 8: NH_4^+ **theoretical content in the solution of NH_4Cl associated with the theoretical volume of HCl for titration and the average results obtained by titration with HCl 1M.**

		NH_4Cl [mg]	NH_4^+ [mg]	Theoretical vol. [ml HCl 1M]
Initial Solution	S1_HCl_1M	50.750	17.236	0,958
	S2_HCl_1M	99.750	33.877	1,882
	S3_HCl_1M	151.250	51.368	2,854

		Measured vol. [ml HCl 1M]	NH_4^+ [mg]
Measured Results	S1_HCl_1M	0.980	17.640±0.624
	S2_HCl_1M	1.893	34.080±0.208
	S3_HCl_1M	2.840	51.120±0.360

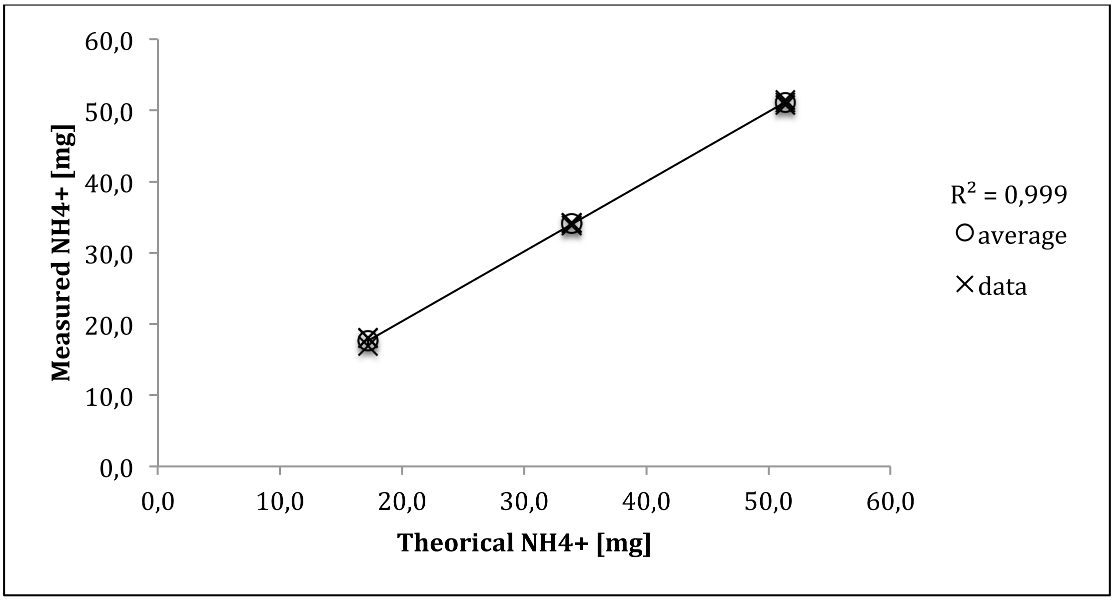


Figure 26: Data from the NH_4^+ quantification with HCl 1M (black crosses) with the regression line, the average values (white points) with the standard deviations.

Table 9: NH_4^+ **theoretical content in the solution of** NH_4Cl **associated with the theoretical volume of HCl for titration and the average results obtained by titration with HCl 10 mM.**

		NH_4Cl [mg]	NH_4^+ [mg]	Theoretical vol. [ml HCl 0,01M]
Initial Solution	S1_HCl_10mM	1.59	0.54	3.00
	S2_HCl_10mM	2.65	0.90	5.00
	S3_HCl_10mM	4.25	1.44	8.01

		Measured vol. [ml HCl 0.01M]	NH_4^+ [mg]
Results	S1_HCl_10mM	2.840	0.511± 0.058
	S2_HCl_10mM	4.873	0.877±0.036
	S3_HCl_10mM	7.667	1.380±0.083

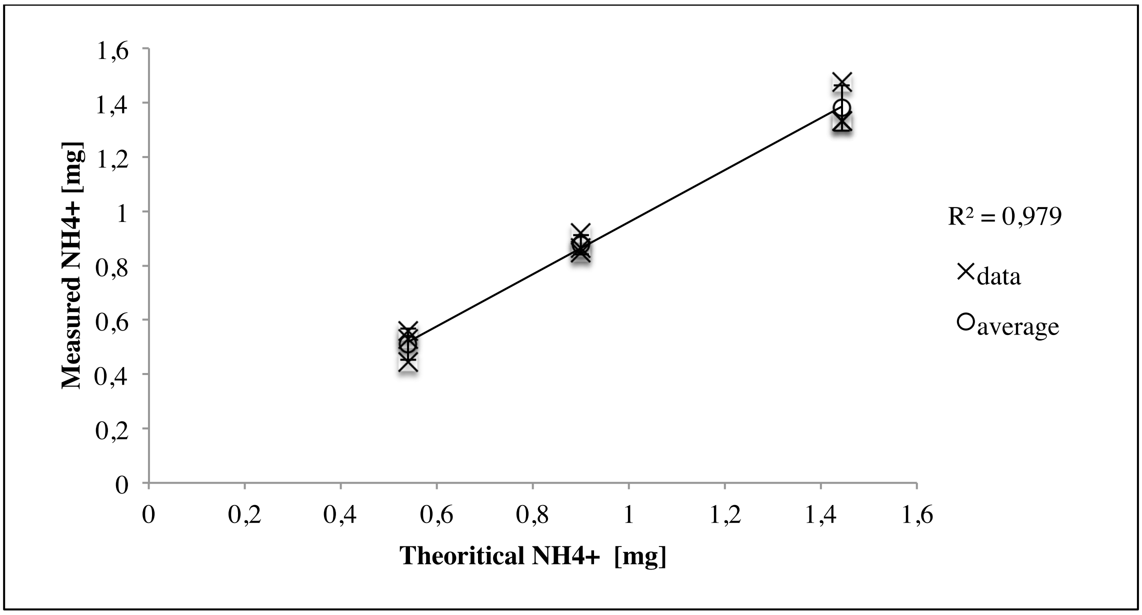
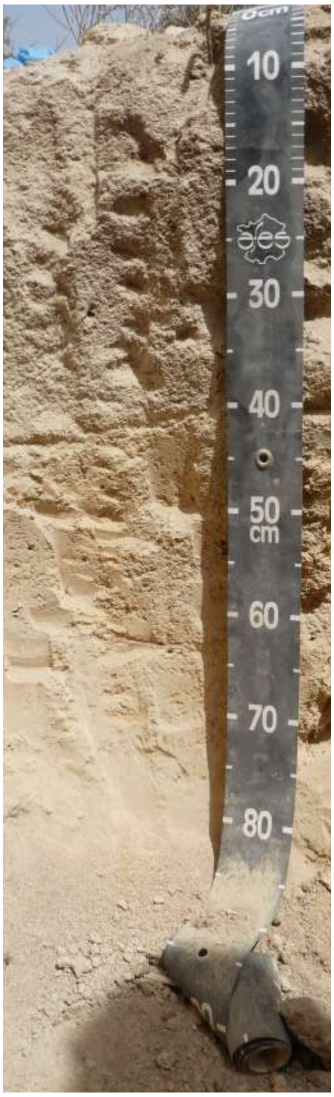


Figure 27: Data from the NH_4^+ **quantification with HCl 10 mM (black crosses) with the regression line, the average values (white points) with the standard deviations.**

3. Soil profile description

The soil profile was located near the village of Koumbia, about 70 km from Bobo-Dioulasso. Dr. M. Traoré described the profile on February 12th 2018 under clear weather conditions with no rain in the past month. The soil moisture regime can be considered as ustic[1], the soil temperature regime was not measured. The profile was situated in a plain landform with a flat slope gradient. The area is used for maize cultivation. The underlying parent material is a granitic formation. According to the BUNASOL the soil is a FLIP or a Lixisol according to the WRB soil classification.



Depth [cm]	Horizon	Description
0-30	E	Homogenous light brown colour due to a higher C_{tot} concentration. Eluvial horizon, leaching of the clays in the underlying horizon. Presence of thin roots. The soil has the matrix of a loamy sand structure.
30-50	Bt1	Gradual colour transition between the eluvial horizon and the argilic horizon from light brown to yellowish colour. Decrease of the C_{org} and increase in clays concentrations. The soil matrix is a sandy clay loam.
50-83	Bt2	No distinct transition with the previous horizon. Homogenous light yellowish colour. Increase of the clays content and decrease of the C_{org} compared to the subsequent horizon. Soil matrix of a clay loam texture.
> 83	R	Granitic parent material

Figure 28: Soil profile description by Dr. Mamadou Traoré on February 12th 2018, site located in the North West of the village of Koumbia in a fallow field. Picture source: Jean-Thomas Cornélis.

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[1] According to the description of the Guidelines for soil description (FAO, 2006)

4 Statistical analyses

4.1 Leaching episodes AV1 analyses

Those tables show the means of each modality for every leaching episodes considered as independent to each other.

Table 10: AV1 results for the P concentrations (mg/L) between the 5 modalities (CBC: co-compost amendment, C: compost amendment, BC: pristine biochar amendment, MBC: mineral loaded biochar amendment, T: control treatment) for every analyses of the leaching (S1 to S8). The mean, standard error and the results of Tukey's HSD test are shown. On the same raw, treatments that do not share the same letters are significantly different (p-value<0.05).

P [mg/L]	CBC			BC			C			MBC			T		
	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey
S5	55,1	55,1	a	10,0	10,0	a	53,6	53,6	a	0,0	0,0	a	23,0	23,0	a
S6	6,8	6,8	a	4,4	4,4	a	5,2	5,2	a	2,8	2,8	a	5,9	5,9	a
S7	2,8	2,8	a	1,3	1,3	a	1,6	1,6	a	0,0	0,0	a	1,4	1,4	a
S8	1,3	1,3	a	0,7	0,7	a	0,7	0,7	a	0,1	0,1	a	0,7	0,7	a

Table 11: AV1 results for the NH_4^+ concentrations (mg/L) between the 5 modalities (CBC: co-compost amendment, C: compost amendment, BC: pristine biochar amendment, MBC: mineral loaded biochar amendment, T: control treatment) for every analyses of the leaching (S1 to S8). The mean, standard error and the results of Tukey's HSD test are shown. On the same raw, treatments that do not share the same letters are significantly different (p-value<0.05).

NH_4^+ [mg/L]	CBC			C			BC			MBC			T		
	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey
S1	15.8	2.3	b	17.4	0.8	b	8.1	1.6	b	155.7	10.2	a	15.0	1.2	b
S2	7.4	0.9	b	30.7	19.4	b	5.3	0.5	b	105.9	17.0	a	4.2	0.8	b
S3	6.4	1.2	b	11.0	2.0	b	4.6	0.1	b	104.1	12.6	a	4.1	1.1	b
S4	0.8	0.8	b	10.3	2.5	b	0.0	0.0	b	61.7	5.1	a	0.0	0.0	b
S5	137.6	51.3	a	65.1	26.1	a	167.8	54.7	a	287.5	210.2	a	83.1	41.9	a
S6	43.8	5.3	a	59.5	8.6	a	61.5	31.7	a	79.9	11.1	a	36.8	3.1	a
S7	18.6	3.9	b	35.9	7.2	b	10.7	7.0	b	103.4	10.4	a	14.9	5.2	b
S8	4.6	3.9	b	23.0	9.5	b	4.5	3.4	b	61.4	5.1	a	6.2	1.3	b

Table 12: AV1 results for the Mg concentration (mg/L) between the 5 modalities (CBC: co-compost amendment, C: compost amendment, BC: pristine biochar amendment, MBC: mineral loaded biochar amendment, T: control treatment) for every analyses of the leaching (S1 to S8). Values without the drainage layer bias. The mean, standard error and the results of Tukey's HSD test are shown. On the same raw, treatments that do not share the same letters are significantly different (p-value<0.05).

Mg [mg/L]	CBC			C			BC			MBC			T		
	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey
S1	101.5	6.9	b	131.0	9.2	ab	62.6	4.4	b	156.6	16.1	a	11.8	1.6	c
S2	13.1	3.2	ab	27.1	7.5	a	18.6	5.9	ab	14.8	5.8	ab	0.0	0.0	b
S3	6.0	3.5	a	0.0	0.0	a	1.3	1.3	a	0.0	0.0	a	0.0	0.0	a
S4	3.3	1.2	b	12.5	3.2	a	1.6	1.0	b	0.9	0.5	b	0.0	0.0	b
S5	0.3	0.3	b	6.6	1.8	a	0.0	0.0	b	0.0	0.0	b	0.0	0.0	b
S6	0.8	0.8	a	6.1	3.5	a	0.0	0.0	a	1.2	1.2	a	1.3	0.7	a
S7	0.0	0.0	a	3.4	1.9	a	0.0	0.0	a	3.0	1.7	a	0.7	0.4	a
S8	0.0	0.0	a	0.0	0.0	a	0.0	0.0	a	0.3	0.3	a	0.0	0.0	a

Table 13: AV1 results for the Ca concentrations (mg/L) between the 5 modalities (CBC: co-compost amendment, C: compost amendment, BC: pristine biochar amendment, MBC: mineral loaded biochar amendment, T: control treatment) for every analyses of the leaching (S1 to S8). Values without the drainage layer bias. The mean, standard error and the results of Tukey's HSD test are shown. On the same raw, treatments that do not share the same letters are significantly different (p-value<0.05).

Ca [mg/L]	**CBC**			**C**			**BC**			**MBC**			**T**		
	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey
S1	480.8	29.6	a	506.2	37.6	a	242.4	17.9	b	587.9	28.1	a	121.3	12.0	c
S2	125.0	31.6	ab	177.1	16.7	a	110.1	22.4	ab	73.5	26.3	b	43.6	11.0	b
S3	37.4	8.8	a	23.7	9.3	a	45.2	10.8	a	1.4	0.9	a	63.9	46.8	a
S4	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/
S5	2.9	2.9	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/
S6	0.0	0.0	a	0.0	0.0	a	30.4	30.4	a	0.0	0.0	a	2.5	2.3	a
S7	0.0	0.0		0.0	0.0	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/
S8	0.0	0.0		0.0	0.0	/	0.0	0.0	/	0.0	0.0	/	0.0	0.0	/

Table 14: AV1 results for the K concentrations (mg/L) between the 5 modalities (CBC: co-compost amendment, C: compost, BC: pristine biochar amendment, MBC: mineral loaded biochar, T: control treatment) for every analyses of the leaching (S1 to S8). Values without the drainage layer bias. The mean, standard error and the results of Tukey's HSD test are shown. On the same raw, treatments that do not share the same letters are significantly different (p-value<0.05).

K [mg/L]	**CBC**			**C**			**BC**			**MBC**			**T**		
	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey	mean	SE	Tukey
S1	59.7	6.5	a	538.6	251.2	a	164.8	114.3	a	133.3	10.6	a	24.6	1.6	a
S2	28.9	5.2	a	396.9	321.6	a	23.0	2.9	a	108.5	18.4	a	15.9	1.6	a
S3	26.0	1.6	b	103.4	13.8	a	17.0	1.6	b	112.1	13.9	a	19.8	8.1	b
S4	125.2	91.1	a	157.6	55.7	a	77.8	75.9	a	143.2	3.8	a	0.3	0.3	a
S5	191.0	48.3	ab	286.7	15.7	a	154.6	51.1	ab	181.0	2.7	ab	67.1	31.7	b
S6	123.9	17.9	b	271.1	23.2	b	50.0	12.7	c	173.1	18.3	a	31.2	3.7	c
S7	99.2	19.9	b	213.8	29.4	a	40.4	10.2	b	194.7	18.0	a	28.6	2.1	b
S8	60.0	13.7	bc	107.6	11.9	ab	29.5	12.3	c	116.5	13.3	a	27.7	12.4	c

4.2 Total leached concentration

Table 15: Mean value of the total leached quantities of nutrients per column, followed by the standard error (SE) and the Tukey's test results for al the treatments (CBC: co-compost amendment, C: compost amendment, BC: pristine biochar amendment, MBC: mineral loaded biochar amendment, T: control treatment).

	K			**Mg**			**Ca**			**P**			**NH_4^+**		
	means	SE	Tukey	means	SE	Tukey	means	SE	Tukey	means	SE	Tukey	means	SE	Tukey
CBC	122.9	11.5	bc	12.0	1.4	bc	54.6	3.5	bc	10.7	4.5	a	36.3	6.6	b
C	323.6	51.3	a	23.6	3.2	ab	77.0	10.1	ab	2.3	1.5	a	40.6	2.4	b
BC	72.3	9.9	c	4.2	0.5	c	21.8	3.4	c	9.0	3.7	a	37.5	11.8	b
MBC	225.5	19.7	ab	30.7	7.6	a	107.7	21.9	a	0.3	0.2	a	165.2	24.1	a
T	37.7	7.3	c	10.3	0.2	bc	70.6	8.1	bc	3.1	2.2	a	27.5	5.4	b

4.3. Regression plant uptake vs total leaching

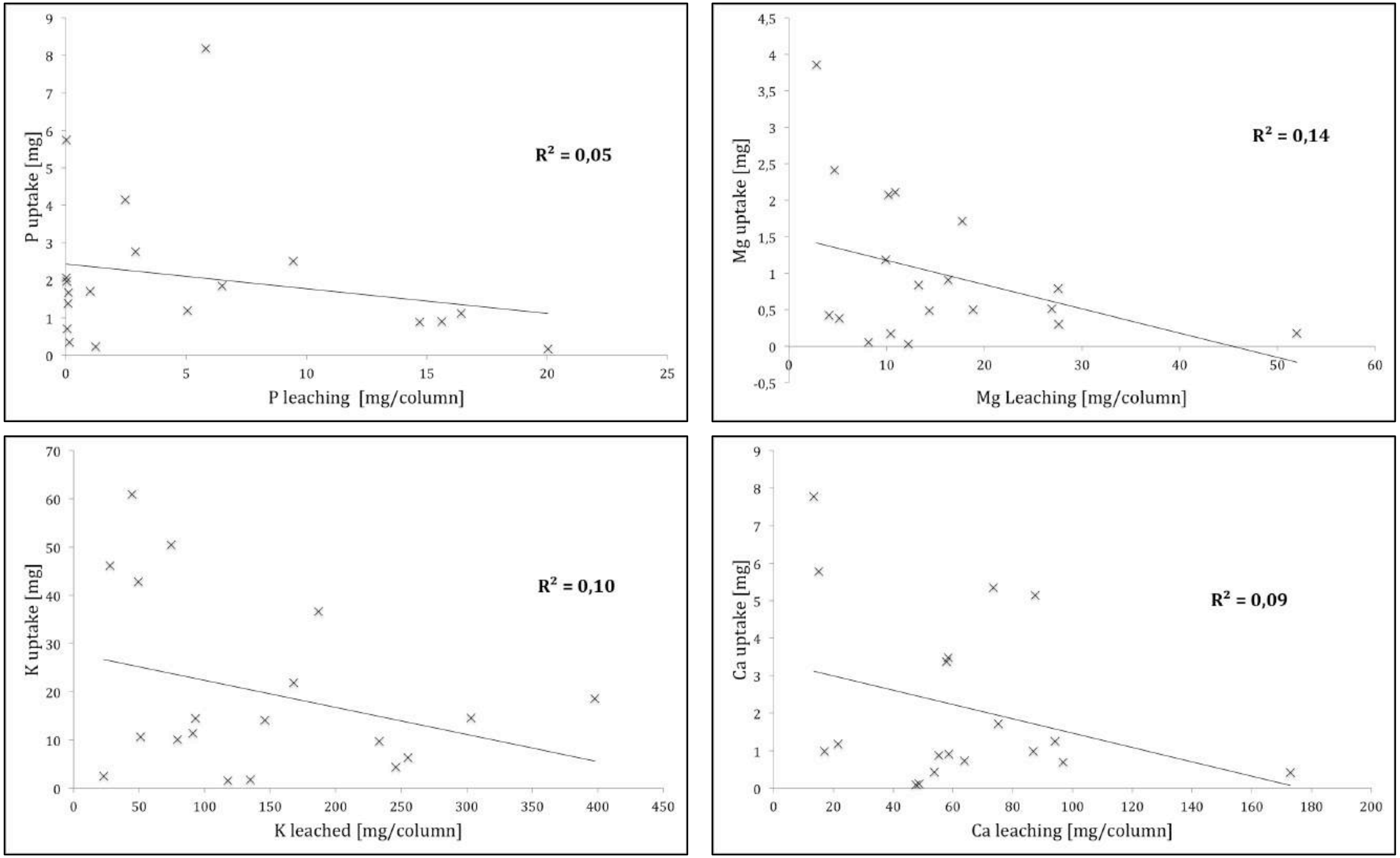

Figure 29 : Regression line between the plants uptake and the total leaching quantities for Calcium, Potassium, Magnesium and Phosphorus

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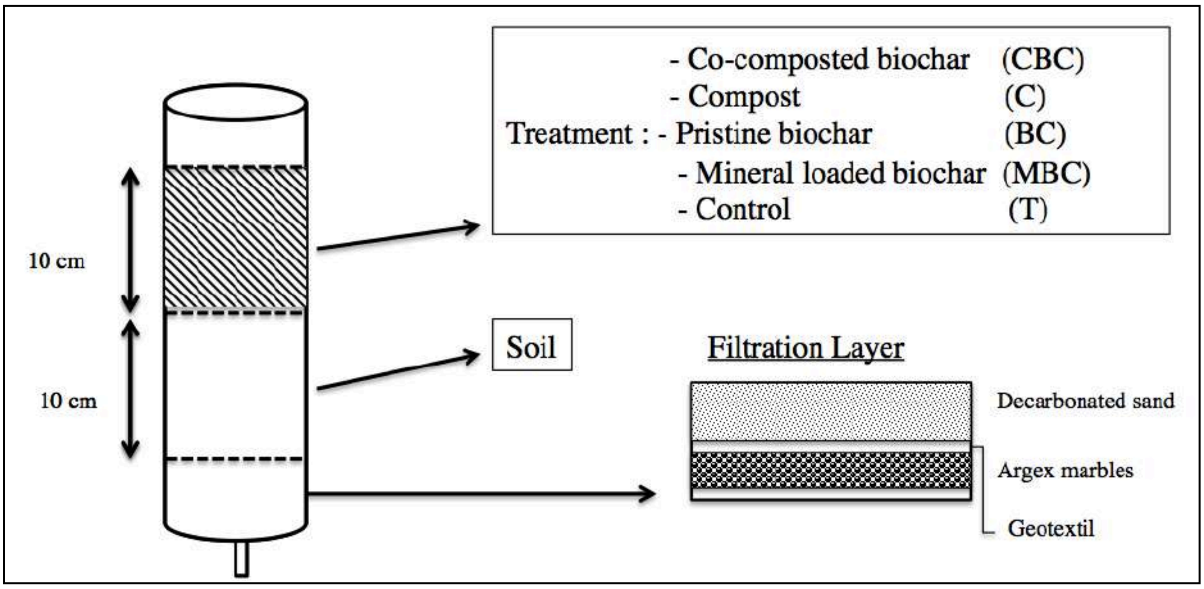
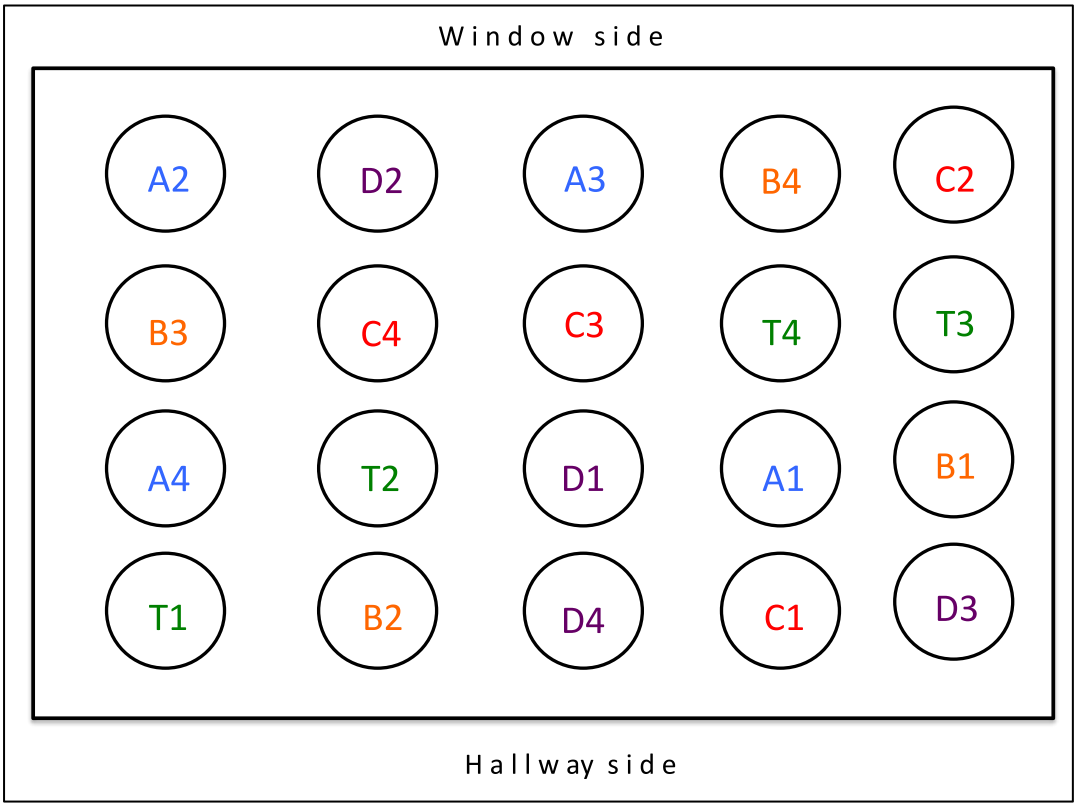
Annexes

Annex 1: Soil columns experiment: (a) details of a column, (b) completely randomized design experimental plan, (c) experimental setup

a)



b)



c)

Annex 2: Soil map for Koumbia area