



# **Production et utilisation du biochar pour l'amendement des sols rouges lessivés tropicaux**

**Thèse**

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## Résumé

Depuis quelques années, la curiosité de la communauté scientifique s'oriente de plus en plus vers la production et l'usage du biochar en agriculture comme amendement. En plus d'être un moyen efficace de valorisation des déchets agricoles et forestiers, il pourrait contribuer à la restauration de la fertilité des oxisols tropicaux et donc au maintien de la productivité des écosystèmes agricoles tropicaux. En retour, cette restauration pourrait contribuer à réduire la pression sur la forêt tropicale, c'est-à-dire la déforestation pour la production agricole. La présente étude a été conduite en plein champ dans la région de l'Ouest Cameroun en Afrique centrale. Elle avait pour objectif de produire, caractériser et tester l'effet de deux biochars, d'origine agricole et forestier, sur les propriétés physico-chimiques d'un oxisol et sur la production et l'équilibre nutritionnel du maïs. Dans un premier temps, il a fallu construire localement un four pyrolytique amélioré par rapport à ce qui se fait actuellement, recyclant les gaz de combustion. Les deux biochars fabriqués à base de résidus locaux (écorce d'eucalyptus et rafles de maïs), avec ce nouveau pyrolyseur amélioré de type « retort » à 300°C, ont été caractérisés (méthodes ASTM, IBI, EBC) et respectaient les normes internationales de biochar. L'expérience au champ avec trois répétitions couvrait 30 parcelles irriguées de 4 m x 4 m chacune, disposées suivant un plan expérimental en split plot. Deux modes de travail du sol, le labour à plat et les sillons-billons, en parcelle principale et en sous parcelle une combinaison aléatoire des cinq traitements contenant l'un ou l'autre des biochars (T2-T3) ou sans biochar (T1), appliqués au début de la première campagne de production uniquement. Le traitement de base dans toutes les parcelles était la dose d'engrais minéral recommandée pour la culture du maïs dans la région à savoir 200 kg NPK ha<sup>-1</sup> + 100 kg N ha<sup>-1</sup>. Le biochar était appliqué à la dose de 15 t ha<sup>-1</sup>. Les propriétés physico-chimiques du sol et des feuilles de maïs ainsi que son rendement sous ces parcelles ont été mesurées, six et douze mois après l'application du biochar. Les résultats ont été analysés par la procédure GLIMIX de SAS suivis du test de comparaison multiple Tukey HSD lorsque nécessaire.

Les résultats suggèrent les conclusions suivantes : les biochars d'écorce d'eucalyptus et de rafles de maïs remplissent bien l'essentiel des critères de définition proposés par le IBI et le EBC pour les biochars. Selon IBI, ce sont des biochars de classe 3 ( $10 \leq \text{Corg} \leq 30$ ). Le

nouveau pyrolyseur pourra servir à produire un biochar de qualité à partir des résidus communs au Cameroun en réduisant les émissions de gaz. L'application du biochar dans nos conditions a eu peu d'effets sur les propriétés physico-chimiques du sol ; cependant, l'augmentation significative du pH (0,3 et 0,5 unités) et du carbone organique du sol (0,4 %) rendent cette technologie acceptable pour le programme global « 4 pour 1000 » initié par la France après la Cop 21. L'analyse nutritionnelle foliaire a révélé une augmentation significative de la teneur en Mg et Ca des plants de maïs dans les parcelles amendées au biochar que non ; le rendement à l'ha du maïs a augmenté de 54 % durant la première période de production et de 51 % durant la seconde dans les parcelles amendées au biochar par rapport à celles non amendées. Cette augmentation de rendement se traduit autrement en une déforestation due à l'agriculture évitée de 25 %. Tous ces résultats indiquent que le biochar pourrait être un outil précieux pour faire face aux enjeux liés à la déforestation et aux changements climatiques dans les régions tropicales humides, ce par une production agricole durable.

## **Abstract**

In recent years, the interest of the scientific community has shifted increasingly towards the production and use of biochar in agriculture as an amendment. In addition to being an efficient means of recovering agricultural and forestry waste, it could contribute to restoring the fertility of tropical oxisols and thus maintaining the productivity of tropical agricultural ecosystems. As a result, this restoration could help in decreasing the pressure on rainforests, that is, deforestation for agricultural production. This field study was carried out in the West region of Cameroon in Central Africa. Its aim was to produce, characterize and test the effect of two biochars from agricultural and forestry origin on the physico-chemical properties of an oxisol and on maize production and maize nutritional equilibrium. Firstly, we constructed locally a retort kiln that improves on the currently-used technology (gas recycling, smoke and pollution reduction, higher biochar yield). The two biochars made from local residues (eucalyptus bark and corn cobs) using this improved kiln at 300 ° C, were characterized using ASTM, IBI and EBC methods. The field experiment included 30 irrigated plots of 4 m × 4 m each, in a split plot design. Two soil tillage modes: flat plowing and furrow-ridges, with three replicates were compared with four biochar treatments, incorporated to soil at the beginning of the first production period. The basic treatment in all plots was the recommended mineral fertilizer rate for maize production in the area: 200 kg NPK +100 kg N. Biochar was applied at 15 t ha<sup>-1</sup>. Maize yield, soil physico-chemical properties and leaf nutritional equilibrium were assessed, six and twelve months after application of the biochar. The results were analyzed using SAS GLIMIX procedure followed by the Tukey HSD multiple comparison test when necessary.

Results suggest the following conclusions: Eucalyptus bark and corncob biochars fulfill most of the criteria definition proposed by IBI and EBC for biochars. According to IBI, these are class 3 biochars ( $10 \leq C_{org} \leq 30$ ). The new pyrolyser can thus be used to produce good quality biochar from common residues in Cameroon with reduced gas emissions. The application of biochar under our conditions has had little effect on the physicochemical properties of the soil; however, the significant increase in pH (0.3 and 0.5 units) and soil organic carbon (0.4 %) makes this technology acceptable for the global program "4 per 1000" initiated by France after Cop 21. Foliar nutritional analysis revealed a significant

increase in the Mg and Ca content of maize plants in biochar amended plots; the yield per hectare of maize increased by 54 % during the first production period and by 51 % during the second in the biochar amended plots compared to the control. This increase in yield is otherwise translated into 25 % avoided deforestation due to agriculture. All these results indicate that biochar could be a valuable tool to face the challenges of deforestation and climate change in the humid tropical zones, through sustainable agricultural production.

## Table des matières

Résumé .....	iii
Abstract .....	v
Table des matières .....	vii
Liste des acronymes, sigles et abréviations .....	x
Liste des tableaux .....	xii
Liste des figures .....	xiii
Remerciements .....	xiv
Avant-propos .....	xvi
Introduction générale .....	1
1. Problématique .....	1
2. Les sols tropicaux rouges lessivés (oxisols) .....	4
2.1 Quelques problèmes agronomiques .....	4
2.2 Potentiel des sols tropicaux à séquestrer le carbone .....	5
3. Biochar et technologies de production .....	5
4. Biochar et production agricole .....	8
4.1 Biochar et propriétés chimiques du sol .....	9
4.2 Biochar et propriétés physiques du sol .....	10
4.3 Nutrition du maïs .....	10
4.4 Biochar et nutrition foliaire .....	11
5. Hypothèse, objectif général, et volets de la recherche .....	12
5.1 Hypothèse de recherche .....	12
5.2 Objectif général de la recherche .....	12
5.3 Volets de la recherche .....	13
Chapitre I .....	14
Quality of biochars made from eucalyptus tree bark and corncob using a pilot-scale retort kiln .....	14
Abstract .....	15
Résumé .....	16
1. Introduction .....	17
2. Material and methods .....	18
2.1 Pilot kiln for pyrolysis .....	18
2.2 Characterization of biochar .....	20

2.2.1	Physical properties .....	20
2.2.2	Chemical properties.....	21
2.2.3	Toxicity related properties .....	23
2.3	Statistical analyses .....	23
3.	Results and Discussion.....	23
3.1	Pilot kiln for pyrolysis .....	24
3.2	General properties of raw material and changes after pyrolysis.....	25
3.3	Physical properties of biochars .....	30
3.4	Chemical properties of biochars .....	33
3.5	Toxicity related properties .....	35
4.	Conclusion.....	36
	Acknowledgements .....	37
	Chapitre II.....	38
	Quantifying the influence of Eucalyptus bark and corncob biochars on the physico-chemical properties of a tropical oxisol under two soil tillage modes .....	38
	Abstract .....	39
	Résumé .....	40
1.	Introduction .....	41
1.1	Biochar and soil physical properties .....	41
1.2	Biochar and soil chemical properties.....	43
2.	Materials and methods.....	45
2.1	Site description and irrigation system.....	45
2.2	Biochar production and characterisation .....	45
2.3	Experimental setup .....	48
2.4	Soil sampling and analysis.....	49
2.5	Statistical analysis.....	49
3.	Results and Discussion.....	54
3.1	Biochar and soil physical properties .....	54
3.1.1	Bulk density and total porosity .....	54
3.1.2	Saturated hydraulic conductivity (Ks), Available water content (AWC) and water retention curve parameters ( $\theta_s$ , $\theta_r$ ).....	55
3.2	Biochar and soil chemical characteristics .....	59
4.	Agonomic implications of the study .....	63



5.	Conclusion.....	63
	Acknowledgements .....	64
	Chapitre III .....	65
	Biochar improves maize nutritional status and yield under two soil tillage modes, on an oxisol, Cameroon .....	65
	Abstract .....	66
	Résumé .....	67
1.	Introduction .....	68
2.	Materials and methods.....	69
2.1	Biochar properties and establishment of field experiment .....	69
2.2	Yield estimation, leaf sampling and analysis.....	70
2.3	Statistical analyses .....	71
3.	Results and Discussion.....	72
3.1	Maize yield in response to biochar application.....	72
3.2	Nutrient concentration and Compositional nutrient diagnosis (CND) of maize leaves .....	73
4.	Conclusion.....	78
	Acknowledgements .....	79
	Conclusions générales, limites et perspectives.....	80
1.	Conclusions générales .....	80
2.	Limites.....	82
3.	Perspectives .....	83
3.1	Perspectives locales .....	83
3.2	Perspectives globales .....	83
	Bibliographie .....	84

## Liste des acronymes, sigles et abréviations

ANOVA	: Analysis of Variance
ASTM	: American Standard Testing Material
AWC	: Available water content
CAEAQ	: Centre d'expertise en analyse environnementale du Québec
CCB	: Corncob biochar
CCNUCC	: Convention Cadre des Nations Unies sur les Changements Climatiques
CEC	: Capacité d'échange cationique
CIS	: Carbone inorganique du sol
CND	: Compositional nutrient diagnosis
CO <sub>2</sub>	: Dioxyde de carbone
COS	: Carbone organique du sol
CVA	: Critical value
DRIS	: Diagnostic and recommendation nutrient diagnosis
EB	: Eucalyptus biochar
EBC	: European biochar certificate
EC	: Electrical conductivity
FAO	: Food and Agricultural Organisation
FASA	: Faculté d'Agronomie et des Sciences Agricoles
FFBC	: Fond Forestier du Bassin du Congo
FP	: Flat plot
FR	: Furrow and ridges
GDT	: Gestion durable des terres
GES	: Gaz à effets de serre
GFW	: Global Forest Watch
GLIMIX	: Generalized mixed linear model
GTZ	: Agence de coopération technique allemande pour le développement
HSD	: Highly significant difference
IBI	: International biochar initiative
IPCC	: Intergovernmental Panel on Climate Change
K <sub>s</sub>	: Saturated hydraulic conductivity
LSD	: Least significant difference
LULUCF	: Land Use, Land Use Change and Forest
MOS	: Matière organique du sol
MPD	: Mean Particle Diameter
PEFOGR	: Programme Élargi de Formation en Gestion des Ressources Naturelles
N-BC	dans le Bassin du Congo
RIFFEAC	: Réseau des Institutions de Formation Forestière et Environnementale d'Afrique Centrale
SAS	: Statistical analysis software

SWC : Soil water content  
USDA : United States Department of Agriculture  
USEPA : United States Environmental Protection Agency  
 $\theta_r$  : Residual water content  
 $\theta_s$  : Water content at saturation

## Liste des tableaux

Table 1 : Chemical properties of corncob biochar (CCB) and eucalyptus biochar (EB) and comparison with the international biochar initiative (IBI) and the European Biochar (EBC) standards.....	26
Table 2: Biochar and soil physical parameters .....	46
Table 3: Biochar and soil chemical parameters before application, 6 and 9 months after application .....	47
Table 4: Summary of methods used for analysing biochar and soil.....	50
Table 5: Analysis of variance for soil physical parameters (degree of freedom and p-values) .....	57
Table 6: Equivalent rate for biochar nutrient and carbon supply, maize needs and recommended fertilizer application rate.....	60
Table 7: Analysis of variance for soil chemical parameters (degree of freedom and p-values) .....	62
Table 8 : Minimum, maximum, average and standard deviation of yield of maize yield during two production periods .....	74
Table 9: Analysis of variance (ANOVA) of maize yield and leaf nutrient concentrations, as influenced by the production period, the treatment and soil management (degree of freedom and p-values).....	75
Table 10 : Optimum leaf nutrient concentration for maize in New Zealand and USA (other studies), compared to high yielding plant leaf nutrient concentrations of the present study.....	76
Table 11 : CND indices values of treated and control plot for each production period....	78

## Liste des figures

Figure 1. Structure of the pyrolyser.....	19
Figure 2. Biochar water sorption by capillary rise under different tensions for corncob biochar (CCB) and eucalyptus biochar. (EB).....	31
Figure 3. Particle size distribution of corncob biochar (CCB) and eucalyptus biochar (EB) .....	32
Figure 4. Capillary rise of CCB and EB under tensions from -0.05 m (very wet) to -1.5 m (wet).....	56
Figure 5. Soil water retention curve as a function of treatment during the first and second production period (values $\pm$ standard error) .....	58

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## **Avant-propos**

La présente thèse est composée de trois chapitres rédigés en anglais sous forme d'articles scientifiques. Pour chaque chapitre, j'atteste être le principal responsable de l'élaboration des travaux de recherche, de la collecte, du traitement et de l'analyse des données ainsi que de la rédaction des articles scientifiques.

**Chapitre I :** Djousse, K.B.M., Allaire, S.E., Munson, A.D., 2017. Quality of Biochars Made from Eucalyptus Tree Bark and Corncob Using a Pilot-Scale Retort Kiln. Waste and Biomass Valorization. doi: 10.1007/s12649-017-9884-2. <https://doi.org/10.1007/s12649-017-9884-2>,

**Chapitre II:** Djousse, K.B.M., Allaire, S.E., Munson, A.D., Quantifying the influence of Eucalyptus bark and corncob biochars on the physico-chemical properties of a tropical oxisol under two soil tillage modes. L'article sera soumis sous peu.

**Chapitre III :** Djousse, K.B.M., Allaire, S.E., Munson, A.D., 2017. Biochar improves maize nutritional status and yield under two soil tillage modes. IJSR, 6 (10), 1-7. <https://www.ijsr.net/archive/v6i10/ART20176493.pdf>

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## **Introduction générale**

### **1. Problématique**

Selon les estimations récentes (Organisation des Nations Unies pour l'alimentation et l'agriculture (FAO), Fonds International de Développement Agricole (FIDA; 2015), la sous-alimentation chronique touche encore un nombre inacceptable de personnes dans le monde; près de 795 millions d'humains. L'écrasante majorité, quelque 780 millions de personnes, vivent dans des pays en développement, particulièrement sous les tropiques. La plupart des personnes vivant dans ce contexte de pauvreté extrême sont tributaires de l'agriculture et des activités connexes. Par ailleurs, la conversion des systèmes naturels en faveur de l'agriculture a occasionné des pertes de 20 à 50 % des stocks de carbone organique du sol (COS), dans le premier mètre de terre (FAO 2007). Depuis, les stocks de COS ont subi une autre réduction du fait de la dégradation des terres en raison des pratiques culturales peu durables telles que le brûlage des champs, des parcours et des résidus de cultures, le labour répété et l'épandage d'engrais sans rétablissement de la matière organique dans le sol. Que ce problème s'exprime en termes de pertes de sols ou de forêts, de disponibilité en eau ou de mauvais rendements des cultures, cet épuisement des terres est attribuable à des pratiques de gestion des terres inefficaces ou non durables, ainsi qu'aux modes d'affectation des terres qui sont inadaptés ou contradictoires (Woodfine 2009). Les activités humaines entravent donc les processus naturels, et cette dynamique est amplifiée par une variabilité croissante du climat.

La forêt dense naturelle au Cameroun couvre la partie sud du pays, avec une superficie estimée de 17,9 millions d'ha (Bikié et al. 2000). Ces auteurs révèlent que plus de la moitié de cette forêt a déjà été exploitée ; cette forêt se dégrade si fortement qu'elle tend même à disparaître par endroits. L'une des causes directes de cette dégradation est l'expansion des pratiques agricoles non durables. Entre 1980 et 1995, presque deux millions d'ha ont été déboisés pour permettre le développement de l'agriculture et l'établissement de communautés humaines (Bikié *et al.*, 2000). En effet, l'agriculture au Cameroun comme dans toute la zone tropicale humide reste dominée par le système traditionnel de la culture sur brûlis. Or ce dernier est de nos jours non durable eu égard à la diminution de la période de jachère. Par ailleurs, l'introduction des engrais minéraux est peu efficace pour des sols

fortement dégradés parce que les nutriments sont très rapidement lessivés. Aussi, les paysans pauvres dans la région ne disposent pas suffisamment de moyens financiers pour se procurer les intrants chimiques nécessaires à l'augmentation de leur production (Fageria et Baligar, 2008; Bekunda *et al.*, 2010). Il en résulte une diminution continue de la fertilité des sols, et par conséquent, une pression importante sur les forêts, entretenue aussi par les besoins en énergie. Cette baisse de fertilité des sols, et l'incapacité des paysans à produire assez d'aliments, freine le développement durable, cause l'insécurité alimentaire et perpétue une situation de pauvreté généralisée.

Les stratégies et pratiques de gestion des terres (GDT) peuvent permettre à ces agriculteurs et à leurs communautés de s'adapter plus facilement aux changements climatiques, d'accroître leur production agricole et énergétique tout en conservant les sols et l'eau et en restaurant leurs ressources productives naturelles. En effet, elles allient les exigences en matière d'agriculture et d'environnement. Dans le domaine de l'agriculture, la GDT comprend l'entretien durable de la productivité des sols. Il faut pour cela conjuguer le traitement de la fertilité des sols (éventuellement l'épandage d'engrais minéraux et organiques) aux mesures de conservation des sols et de l'eau, comme le billonnage selon les courbes de niveau, les cultures en terrasses, les billons cloisonnés ou encore l'apport d'une couverture végétale au moyen du paillis (Woodfine 2009). Vu l'absence d'une seule « solution miracle » aux problèmes de dégradation des terres et de faible productivité, le choix des technologies et des approches adéquates de GDT pour une zone particulière sera déterminé par : 1) les qualités et caractéristiques des ressources foncières locales ; 2) les exigences en GDT du mode d'affectation des terres à exploiter ; et 3) le contexte socioéconomique et les priorités des usagers des terres (Woodfine 2009).

En Afrique subsaharienne, la GDT paraît donc comme le point d'entrée incontournable pour accroître la résilience et la productivité des ressources foncières face au changement climatique. Selon Guo et Gifford (2002), l'activité la plus importante à réaliser au cours des prochaines décennies pour atténuer le changement climatique en Afrique est d'accroître la quantité de carbone séquestrée biologiquement dans la biomasse et la matière organique du sol. En effet, l'augmentation du COS est bien connue pour ses bienfaits multiples sur les sols (Lal 2004), dont : i) une fertilité accrue grâce à la rétention des nutriments ; ii) des taux

croissants d'infiltration des eaux de pluie ; iii) une capacité accrue de rétention de l'eau; enfin iv) de meilleures conditions pour la faune terrestre et autres organismes du sol comme les vers de terre et les termites. Ainsi, en stabilisant une structure terrestre largement améliorée, on accroît « la résilience des terres ».

L'une des alternatives de GDT est l'usage du biochar en agriculture (Woodfine 2009). En effet, les travaux de recherche récents révèlent que les matières carbonisées (biochar) obtenues à partir de la combustion de matière organique en absence d'oxygène (pyrolyse) sont à l'origine du maintien des propriétés agronomiques exceptionnelles, semblables à celles décrites ci haut (fortes quantités de carbone, de nutriments et meilleure capacité de rétention en eau) dans les sols anthropiques (terra preta, ou terres noires en portugais) du bassin de l'Amazonie au Brésil (Glaser et al. 2002; Lehmann et al. 2002; Sohi et al. 2010). L'usage du biochar en agriculture paraît comme une pratique de GDT prometteuse pour l'Afrique, car elle relie les questions d'amélioration des terres dégradées, d'augmentation du rendement des cultures, d'approvisionnement en énergie, d'atténuation des effets du changement climatique et de développement rural. Il est adapté à une vaste gamme de situations, en partant des ménages, des fermes ou villages qui pourraient en produire pour leurs propres besoins jusqu'aux grandes installations.

Au Cameroun, les résidus des industries de première transformation du bois estimés à 1 752 000 m<sup>3</sup>/an (GTZ 2006) restent encore peu ou pas exploités. Ce potentiel s'est probablement accru ces dernières années avec la création progressive de petites unités de transformation suite à l'interdiction d'exportation des grumes de bois. À cela s'ajoute les sous-produits d'exploitation forestière directe, et des différents déchets générés au cours de la production. Les résidus forestiers (cimes, branches, souches, racines, coursons) abandonnés dans les parcs à bois représentent environ 50% du volume sur pied ; soit 2,5 millions de m<sup>3</sup> an<sup>-1</sup>. Tous ces résidus en plus des résidus agricoles constituent un potentiel énorme pour la production du biochar. Il est important de noter que, pour le moment, seules les ressources en biomasse qui peuvent être obtenues de manière durable, c'est à dire sans compromettre la sécurité alimentaire, la biodiversité ou la conservation des sols, sont recommandées comme matière première pour produire le biochar (GTZ 2006). Cependant, de nombreuses questions restent à ce jour peu couvertes sur les techniques de production, les modes d'application, les

mécanismes d'action dans le sol et l'influence sur la nutrition des plantes (Steiner et al. 2007; Verheijen et al. 2010; Atkinson et al. 2010). Ce travail vise donc à contribuer à l'amélioration des connaissances techniques dans chacun de ces quatre aspects dans un environnement tropical africain.

## **2. Les sols tropicaux rouges lessivés (oxisols)**

### **2.1 *Quelques problèmes agronomiques***

Le sol peut être défini comme une « entité naturelle, superficielle et souvent meuble résultant de la transformation au contact de l'atmosphère et des êtres vivants, d'un matériau minéral issu le plus souvent d'une roche sous-jacente, sous l'influence de processus physiques, chimiques et biologiques » (Girard et al. 2011). L'association de ce matériau minéral avec les matières organiques du sol (MOS) issues des êtres vivants va notamment former des complexes organo-minéraux dans les sols. Un sol naturel est composé de trois phases. La phase solide formée d'un mélange d'éléments minéraux et d'éléments organiques qui constituent le squelette ou la matrice du sol. Les éléments minéraux fins (argiles) et la matière organique décomposée (humus) sont liés au complexe argilo-humique du sol, la phase liquide constituée par l'eau retenue dans les pores du sol et, la phase gazeuse qui remplit les pores du sol (Musy & Soutter 1991). Les principaux sols rencontrés dans les tropiques sont : Les Oxisols, les Alfisols et Ultisols, les Vertisols, les Andisols, les Inceptisols, les Spodosols et les Entisols (USDA 2014). Nous nous intéresserons particulièrement aux oxisols, car ils y sont les plus abondants et les plus problématiques.

Selon Soil Survey Staff, (2010), le terme « ox » de oxisols indique les sols les plus vieux et altérés dans la taxonomie. Les oxisols (ferralsols, selon la classification de la FAO et sols ferrallitiques selon la classification française) sont dominés par les oxydes de fer et d'aluminium, qui sont très infertiles et difficiles à gérer du point de vue agronomique et environnemental. Les processus d'hydrolyse, d'hydratation, de dissolution, d'oxydation, de désilication, d'acidification et de lessivage sont très intenses dans ces sols. Ils sont donc constitués essentiellement de minéraux secondaires (néoformés). Leur pH est très bas, ils présentent des profils assez profonds et de fortes teneurs en aluminium. La fraction argileuse (<2 mm) de ces sols est majoritairement constituée de phyllosilicates de type 1:1 comme la kaolinite, ainsi que d'oxydes et hydroxydes de fer et d'aluminium. Ceux-ci présentent une

faible habileté à retenir les éléments nutritifs. À cause du faible pH de ces sols, l'aluminium, qui est toxique à de nombreuses espèces végétales, est rendu mobile et les sesquioxydes fixent le phosphore du sol, le rendant indisponible pour les plantes (Sanchez et Logan, 1992). Les argiles et les sesquioxydes forment très souvent des agrégats assez stables qui assurent un drainage excellent de ces sols, favorisant malheureusement aussi le processus de lessivage. Lorsque des obstacles sont présents dans le sol, les sesquioxydes et les argiles lessivés s'accumulent et se transforment très souvent en latérite. Celle-ci limite l'oxygénation des racines ainsi que leur développement.

## ***2.2 Potentiel des sols tropicaux à séquestrer le carbone***

Les écosystèmes terrestres, en retenant le carbone dans la biomasse vivante, dans les matières organiques en décomposition et dans les sols minéraux, jouent un rôle important dans le cycle global du carbone. Le stock de carbone dans les sols est d'environ deux fois supérieure à celui de l'atmosphère et trois à quatre fois supérieur à celui de la végétation (Griggs & Nogueir 2002; Denman et al. 2007). En plus de sa capacité de stockage du carbone de l'atmosphère, le sol joue un second rôle important dans les flux globaux des autres gaz à fort potentiel d'effet de serre, car il émet et absorbe le méthane et l'oxyde nitreux. Lal (2001) affirme que la teneur en carbone des sols tropicaux varie entre 308-506 Pg pour le carbone organique (COS) et 149-218 Pg pour le carbone inorganique du sol (CIS); soit une contribution de 32 % au stock de carbone du sol mondial évalué à 1550 Pg. Il estime sa répartition comme suit : 201 Pg dans les sols en Afrique (40,5 %), 198 Pg en Amérique tropicale (39,9 %) et 97 Pg en Asie tropicale. La contribution des oxisols au stockage du COS est de 157 Pg soit 38,5 % du stock total dans les sols tropicaux. Les oxisols étant très pauvres en matière organique auraient donc un grand potentiel de stockage de carbone s'ils sont bien gérés.

## **3. Biochar et technologies de production**

Le biochar se définit comme le produit de la thermo-dégradation de la matière organique en milieu pauvre en oxygène, destiné à un usage agricole (Lehmann & Joseph 2009). Ce processus de transformation est dénommé pyrolyse. De nombreux modèles de fours à pyrolyse existent pour la production de biochar, chacun aboutissant à l'obtention d'une gamme de produits en proportions diverses (biogaz, biohuile et biochar). Verheijen *et al.*

(2010) distingue trois types de pyrolyses : rapide, intermédiaire et lente, cette dernière parfois appelée carbonisation (à cause de la concentration de la fraction de carbone dans la matière).

La pyrolyse rapide se fait généralement avec des dispositifs améliorés du type « fluidized bed reactor » qui polluent très peu car ils recyclent les gaz et condensent les fumées. La biomasse est portée très rapidement ( $< 1$  s) à une température comprise entre 400 et 700 °C en absence d'oxygène. Pour cela, la biomasse doit être réduite en particules  $< 2$  mm, ce qui requiert une quantité importante d'énergie pour le pré conditionnement (Cummer & Brown 2002). Cette technologie produit seulement 15-25 % de biochar et 60-70 % de bio-huiles après condensation des vapeurs de pyrolyse (Bridgwater & Peacocke 1994; Czernik & Bridgwater 2004; Mohan et al. 2006). Le biochar obtenu est très riche en condensés aromatiques. Ceci le prédispose à être mieux séquestré dans le sol eu égard à la nature récalcitrante de ses composés. La pyrolyse intermédiaire requiert un pré conditionnement similaire au précédent, seulement le temps de séjour dans le four est plus long (10-20 secondes) et la proportion de bio huile obtenue est supérieure à celle de la pyrolyse lente.

La pyrolyse lente se fait soit avec des fours traditionnels ou des fours modernes. Les fours traditionnels « traditional charcoal kiln » ne recyclent aucun sous-produit de la thermo-dégradation, ils sont moins efficaces énergétiquement et produisent des émissions polluantes (Kammen & Lew 2005). Ces modèles de fours sont fortement déconseillés surtout dans un contexte de valorisation de la biomasse sous forme de biochar. Avec les fours modernes à pyrolyse lente, les matières volatiles produites lors de la thermo-dégradation de la biomasse, sont soit capturées et condensées pour être réutilisées comme produits chimiques (huiles pyrolytiques), directement brûlées, soit pour sécher la matière, ce qui améliore l'efficacité du four, ou pour produire de l'électricité à travers des centrales thermiques. La quantité de biochar produite est plus importante et ce biochar contient plus de groupes fonctionnels du type C=O et C-H qui peuvent servir éventuellement de sites d'échange pour les nutriments du sol après oxydation (Glaser et al. 2002). Mieux encore, ils sont plus faciles à manipuler et requièrent moins de contrôle, présentent des caractères organiques incluant des structures de type aliphatiques et cellulosiques. Eu égard à tous ces détails, nous avons opté pour l'utilisation de la pyrolyse lente dans cette étude.

McLaughlin et al. (2009) identifient quatre fractions importantes qui constituent le biochar : l'humidité, les cendres, les matières labiles et les matières récalcitrantes. Selon leurs travaux, le biochar pourrait absorber jusqu'à trois fois son poids sous forme d'humidité; des résultats similaires ont aussi été obtenu par Allaire et al. (2015). Le biochar à base de bois (matière ligneuse) est généralement faible en cendre (< 1 %), alors que celui issu de la biomasse riche en minéraux comme les résidus agricoles en contient jusqu'à 24 % (Amonette & Joseph 2009). Le biochar produit à base des déjections animales peut contenir jusqu'à 45 % de cendres (Koutcheiko et al. 2007). Les cendres du biochar sont constituées essentiellement de calcium (Ca), fer (Fe), magnésium (Mg), sodium (Na), potassium (K), phosphore (P), silice (Si) et d'aluminium (Al) (Amonette & Joseph 2009). Les matières labiles font référence à la fraction du biochar qui s'altère de manière abiotique ou biotique quelques jours à quelques semaines suivant son incorporation dans le sol. Cette fraction est importante, car sa décomposition peut entraîner l'immobilisation de l'azote par la biomasse microbienne, suite à une augmentation du rapport C/N. Cette immobilisation est souvent suivie d'une baisse temporaire de rendement des cultures (Rondon et al. 2007; Asai et al. 2009; Blackwell et al. 2010). La matière récalcitrante, essentiellement le carbone avec sa structure fortement aromatique, constitue la grande proportion du biochar et lui garantit sa stabilité chimique dans le sol (Sohi et al. 2010).

La diversité des méthodes de production ainsi que des biomasses utilisées constituent un défi pour la valorisation agronomique des différents biochars (Schmidt et al. 2000; Lehmann & Joseph 2009). En effet, la qualité finale du biochar dépend de la biomasse utilisée et des conditions de fabrication, tels que la température et le temps de séjour dans le four. La manipulation de l'un de ces trois facteurs permettrait au producteur d'orienter la pyrolyse en fonction des objectifs d'usage visés. Par ailleurs, certains biochars peuvent soit stimuler ou inhiber le développement de la faune du sol (Liesch et al. 2010); soit stimuler ou inhiber la germination et la croissance de la plantule chez certaines espèces (Chan et al. 2008; Free et al. 2010; Van Zwieten et al. 2010). D'où la nécessité pour une meilleure compréhension des mécanismes d'action du biochar, de préciser pour chaque expérience ses caractéristiques physico-chimiques ainsi que les détails de fabrication (biomasse utilisée, température de pyrolyse, le taux d'application et la méthode d'application) (Warnock et al. 2007; Tomlinson et al. 2012). Mieux encore, ceci permettrait de mettre sur pied des directives de production



du biochar en fonction des besoins spécifiques. L'Initiative Internationale pour le Biochar (IBI) recommande la réalisation des tests préliminaires de toxicité en rapport avec ces deux aspects dans le cadre de la caractérisation du biochar.

#### **4. Biochar et production agricole**

Il est admis de nos jours que les terres très fertiles de l'Amazonie (Terra Preta de Indio) résultent de l'enfouissement des résidus de toutes sortes dont un certain pourcentage de résidus carbonisés (Lehmann *et al.*, 2003). Les rendements élevés obtenus sur ces terres ont poussé les chercheurs à évaluer le biochar comme amendement pour améliorer les propriétés des sols agricoles. Plusieurs publications récentes rapportent que l'utilisation du biochar peut influencer: la croissance et les rendements des plantes et leur état sanitaire (Glaser *et al.* 2001; Glaser *et al.* 2002; Lehmann & Joseph 2009; Elad *et al.* 2010); la diminution des gaz à effet de serre dégagés par le sol (Jeffery *et al.* 2011; Spokas *et al.* 2011); l'établissement des symbioses ou des associations bénéfiques (mycorhizes, rhizobiums); l'activité enzymatique des sols (O'Neill *et al.* 2009; Kolton *et al.* 2011). Les effets bénéfiques du biochar semblent être plutôt indirects et reliés à sa capacité à stimuler et augmenter la biodiversité microbologique du sol, à améliorer les propriétés physiques du sol et limiter le lessivage des éléments nutritifs. Cependant, bon nombre de ces travaux soulignent la nécessité de multiplier les recherches sur le biochar issu de différents types de biomasses ainsi que leur application dans de nombreuses régions du globe.

Plusieurs études ont déjà été rapportées sur la culture du maïs avec des biochars de diverses origines. Dans certaines de ces études, l'application du biochar a engendré une augmentation du rendement de 2,2 t ha<sup>-1</sup> par rapport au témoin (Van Zwieten *et al.* 2010; Sukartono *et al.* 2011), dans d'autres une augmentation de 20% à 140 % par rapport au témoin (Oguntunde *et al.* 2004; Crane-Droesch *et al.* 2013). Des résultats positifs ont aussi été observés avec la culture du blé (Blackwell *et al.* 2010), du soja (Husk & Major 2011) et d'autres légumineuses (Sovu *et al.* 2012). Cependant il y a également quelques études qui révèlent soit une baisse ou une constance de rendement (Gaskin *et al.* 2010; Jones *et al.* 2011). Des résultats contrastants similaires à ceux cités plus haut ont été obtenus avec la culture du riz (Asai *et al.* 2009; Petter *et al.* 2012). Toutes ces études ont généralement été conduites soit en serre, soit dans des systèmes de culture mécanisés. Cependant, dans les systèmes traditionnels de

la plupart des pays en voie de développement, les opérations de travail du sol sont généralement caractérisées par des intrants énergétiquement faibles. Les houes dans le cas du travail du sol et la machette pour le désherbage sont les principaux outils manuels en Afrique centrale. L'emploi de la houe manuelle pour préparer la terre en billons, en tas ou en monticules est une méthode très répandue liée au labour primaire. Dans ce cas, une partie de plus en plus grande du volume total du sol est labourée. Du sol de surface est donc collecté ce qui provoque une augmentation de volume du sol pour les semis et le lit racinaire. Le sol du lit de semis est enrichi par la matière organique provenant des mauvaises herbes ou des résidus tandis que l'aération et le drainage sont favorisés. Ce système est encore plus important dans les régions de hautes terres comme celles de l'ouest Cameroun où la topographie constitue un frein à la mécanisation et donc au labour conventionnel (labour à plat avec herbes et charrues). Le biochar, utilisé seul ou en combinaison avec d'autres résidus agricoles a jusqu'à ce jour fait l'objet de peu ou pas d'études en rapport avec le billonnage manuel caractéristique des pays Africains. Or, la manière de travailler le sol peut modifier entre-autres sa température et sa teneur en eau (Curtin et al. 2000; Al-Kaisi & Yin 2005). Ces deux paramètres influencent de manière indirecte les taux d'émissions de CO<sub>2</sub> du sol (Bajracharya et al. 2000; Amos et al. 2005) et donc la perte du carbone du sol. Peu ou pas d'études ont jusqu'à présent été faites sur la réponse de ces systèmes de culture locaux à l'amendement au biochar. Ceci pourrait constituer une barrière à l'appropriation de la technologie par les paysans. Mieux encore, il serait nécessaire de combiner des études de diagnostic foliaire des plantes cultivées sur sols amendés au biochar afin de mieux interpréter la réponse agronomique de ce dernier.

#### 4.1 *Biochar et propriétés chimiques du sol*

Lorsqu'incorporé dans le sol, le biochar pourrait modifier certaines propriétés chimiques des sols telles que le pH et le rapport C/N. Ceci aurait des implications importantes sur la CEC et la disponibilité en macroéléments nutritifs majeurs (N, P, K, Mg, Ca) du sol (Lehmann et al. 2003; Verheijen et al. 2010). Plutôt qu'un amendement, le biochar se comporterait comme un restructurateur du sol et peut-être comme un catalyseur, via des mécanismes d'action encore mal compris (Steiner et al. 2007). Sa contribution relative à l'altération de ces propriétés chimiques et sa stabilité dans le sol sous différents modes de gestion des terres

reste peu documentée. Aussi, les doses utilisées restent encore assez variables. Lehmann *et al.* (2002) préconisent la dose de 11 t ha<sup>-1</sup>, qui correspond au taux attendu à la suite d'une culture sur brûlis dans une forêt secondaire moyenne poussant sur un sol ferrallitique de l'Amazonie centrale. Cependant, plusieurs taux différents ont été déjà testés avec différentes cultures.

#### 4.2 *Biochar et propriétés physiques du sol*

Le biochar est hautement poreux, et donc son application au sol améliorerait une gamme de propriétés physiques du sol, y compris la porosité totale, la distribution des pores, la masse volumique apparente, la teneur en humidité du sol, la teneur en eau disponible, l'infiltration et la conductivité hydraulique (Major *et al.* 2010; Atkinson *et al.* 2010). Cependant, il existe peu d'études *in situ* qui confirment que l'application du biochar améliore de façon importante les propriétés physiques des sols agricoles (Sohi *et al.* 2009; Shackley *et al.* 2010). En outre, les mécanismes ou processus par lesquels le biochar peut influencer la distribution de la taille des pores du sol n'ont pas été clairement établis ou démontrés (Verheijen *et al.* 2010). S'il est déjà admis que l'ajout du biochar serait plus bénéfique sur les sols sableux que les sols argileux, il reste que peu d'études se sont jusqu'à présent focalisées sur l'influence des propriétés et du type de biochar sur la courbe de rétention en eau du sol et le mouvement de l'eau, sous différents modes de travail du sol. Par exemple, le billonnage présente des avantages pour la gestion de l'infiltration, mais aussi pour l'établissement de la culture à cause du faible compactage du sol dans le billon. Par contre, cette faible densité peut entraîner un séchage plus rapide du sol autour de la semence et donc augmenter les risques d'échecs pour les cultures en conditions sèches.

#### 4.3 *Nutrition du maïs*

Pour boucler son cycle, le maïs mobilise approximativement 240 kg ha<sup>-1</sup> d'N, 90 kg ha<sup>-1</sup> de P<sub>2</sub>O<sub>5</sub>, 270 kg ha<sup>-1</sup> de K<sub>2</sub>O, 40 kg ha<sup>-1</sup> de Mg, 60 kg ha<sup>-1</sup> de CaO. Ces quantités peuvent varier en fonction du type de sol et du rendement escompté. Plus de 50 % de l'azote, du phosphore, du magnésium et plus de 80 % du potassium sont absorbés avant la floraison mâle (YARA, 2017a). L'azote (N) est une composante principale des protéines, et par conséquent des tissus des plantes. La principale source d'azote pour les terres non fertilisées est la matière organique, cette dernière est décomposée par les bactéries et les champignons, qui

transforment l'azote organique en ammoniacque et éventuellement en nitrate (NO<sub>3</sub>-), absorbable par les racines des plantes. Le phosphore (P) joue un rôle important dans la formation des fruits et des grains ; Il est indispensable même en faible quantité aux stades précoces pour assurer un bon démarrage de culture, en stimulant l'enracinement et en accélérant l'émergence des premières feuilles. Il est présent dans le sol sous trois formes : la forme libre, la forme stable et la réserve minérale.

Le potassium (K) est un élément qui influence l'absorption des autres éléments. Il contribue à favoriser la floraison et le développement des fruits. Comme l'azote, il renforce le développement de la culture et un équilibre est nécessaire entre la nutrition azotée et la nutrition potassique. Le potassium dans le sol est généralement sous une forme facilement absorbable par la plante. Absorbé dans des quantités supérieures à l'azote, le potassium se localise principalement dans les feuilles et les tiges et finalement la récolte de grain n'exporte qu'une faible part du potassium mobilisé. A l'inverse, le maïs fourrage exporte des quantités importantes de potasse. Prélevés en très faibles quantités, les oligo-éléments demeurent nécessaires pour assurer la croissance de la culture et atteindre le rendement visé. Contenus principalement dans les feuilles et les tiges, ils sont plus fortement exportés lors d'une récolte en ensilage (YARA, 2017b). Les oligo-éléments prélevés en plus grandes quantités par le maïs sont le fer (1900 g ha<sup>-1</sup>) et le manganèse (340 g ha<sup>-1</sup>). Le magnésium, le soufre et le fer participent à l'activité photosynthétique et maintiennent une bonne croissance indispensable pour atteindre des rendements élevés. Le calcium contribue à la vigueur de la culture, à sa résistance à la sécheresse, protège les racines et augmente la résistance des feuilles aux maladies.

#### 4.4 *Biochar et nutrition foliaire*

La satisfaction des besoins minéraux d'une culture dépend de l'aptitude du sol à fournir les nutriments en quantité suffisante et suivant la demande de la culture, en termes d'intensité et de répartition (cycle, enracinement). Les termes de l'offre et de la demande sont en partie contrôlés par les conditions de milieu (conditions hydriques, acidité, état physique du sol). Ils sont également très interdépendants. La démarche du diagnostic nutritionnel repose sur la réponse non linéaire de la croissance aux consommations minérales. Le diagnostic foliaire est donc un outil complémentaire à l'analyse de sol et permet d'ajuster les recommandations

de fertilisation. Différentes techniques de diagnostic foliaire ont été successivement élaborées dans le but d'étendre les limites de cette technique. Nous présenterons ici les trois principales généralement utilisées. La méthode CVA (critical value) est basée sur l'idée que chaque nutriment peut être caractérisé au sein d'une plante par une concentration optimale et par une concentration critique minimale, elle n'intègre donc pas la notion d'équilibre nutritionnel. En adéquation avec une notion de dépendance entre nutriments, la méthode bivariée dite « diagnostic and recommendation nutrient diagnosis » (DRIS) a été développée (Beaufils, 1973). Elle permet de déterminer pour chacun des nutriments considérés, des index calculés selon un rapport entre des ratios de nutriments de référence et des ratios de nutriments observés. Mais cette dernière considère uniquement les nutriments analysés et non la totalité des nutriments constitutifs. Parent et Dafir, (1992) présentent une méthode multivariée appelée CND (compositional nutrient diagnosis) permettant de connecter et d'offrir les mêmes avantages que les méthodes CVA (méthode univariée) et DRIS (méthode bivariée) réunies, en maintenant une symétrie dans la formule de calcul des index nutritionnels. Les méthodes DRIS et CND ont en commun le principe de classement des nutriments du plus limitant au moins limitant afin de renseigner sur l'équilibre nutritionnel global des nutriments.

## **5. Hypothèse, objectif général, et volets de la recherche**

### **5.1 *Hypothèse de recherche***

L'hypothèse principale de cette recherche est que le rendement de la culture de maïs sur sols ferrallitiques pourrait être amélioré sous deux pratiques culturales locales en associant à la fumure minérale habituelle (NPK) deux types de biochars. Cette amélioration est associée aux propriétés physiques et chimiques spécifiques du biochar et son interaction avec le sol.

### **5.2 *Objectif général de la recherche***

Ce projet de recherche se propose donc de contribuer à l'amélioration des connaissances sur la production et l'utilisation du biochar en agriculture tropicale comme moyen de lutte et d'adaptation aux changements climatiques. Il se focalise sur les problématiques fondamentales en rapport avec les techniques de production, les modes d'application, les mécanismes d'action dans le sol et sur la plante.

### 5.3 *Volets de la recherche*

Trois grands volets de recherche représentant les trois chapitres principaux de cette thèse.

Le volet 1 (chapitre 1) est consacré à la production et la caractérisation de deux biochars dont un d'origine agricole et l'autre d'origine forestière par un four pyrolytique amélioré, recirculant les gaz de pyrolyse. La question principale de recherche ici est celle de savoir comment produire localement des biochars qui respectent les normes IBI et Certificat Européen pour le Biochar (EBC). Nous avons conçu et fabriqué un pyrolyseur de type « retort » qui présente l'avantage de recirculer les gaz de synthèse produits lors du processus de pyrolyse. Les tests réalisés étaient en rapport soit avec les propriétés de base du biochar, sa toxicité et ses propriétés plus détaillées plus détaillée, ce conformément aux recommandations de l'IBI.

Le volet 2 (chapitre 2) a pour objectif de quantifier l'influence des biochars sur les propriétés physiques et chimiques d'un oxisol sous deux modes de culture : le labour à plat et les sillons-billons. La parcelle expérimentale a été divisée en 3 blocs recevant chacun en parcelle principale de manière totalement aléatoire les 2 modes de travail du sol et en sous parcelle un arrangement aléatoire des 5 traitements. Soit un total de 30 parcelles de 16 m<sup>2</sup> chacune avec un mini système d'irrigation. L'hypothèse de recherche était que l'ajout du biochar à l'oxisol améliorera ses propriétés physiques et chimiques. Des échantillons de sol collectés avant application du biochar, puis 6 et 12 mois après ont été analysés au laboratoire.

Dans le volet 3 (chapitre 3), il était question d'évaluer l'effet de deux biochars sur l'équilibre nutritionnel et le rendement des plants de maïs sous deux modes de labour du sol. Nous avons émis l'hypothèse que l'application du biochar à 15 t ha<sup>-1</sup> va induire un meilleur équilibre nutritionnel des plants du maïs et une augmentation significative de son rendement au cours des deux premières périodes de production. Le même dispositif expérimental décrit au volet 2 a été utilisé. Afin d'évaluer l'équilibre nutritionnel, des échantillons composites de feuilles des plants de maïs ont été collectés à la floraison pendant la première et seconde saison de production, puis analysés. Vingt et cinq épis de maïs récoltés sur une surface équivalente de 6 m<sup>2</sup> ont été égrainés séchés et pesés pour l'évaluation du rendement.



## **Chapitre I**

### **Quality of biochars made from eucalyptus tree bark and corncob using a pilot-scale retort kiln <sup>1</sup>**

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<sup>1</sup> Version intégrale d'un article publié : Djousse, K.B.M., Allaire, S.E., Munson, A.D., 2017. Quality of Biochars Made from Eucalyptus Tree Bark and Corncob Using a Pilot-Scale Retort Kiln. Waste and Biomass Valorization. doi:10.1007/s12649-017-9884-2



## **Abstract**

In many developing countries, traditional earth-mound kilns are still the principal technology for biochar production. The present study focused on the production and characterization of two biochars from common agricultural and forestry residues in Cameroon: corncob and eucalyptus tree bark. A pilot-scale retort kiln, made from local material, was constructed for biochar production. Its production efficiency varied between 33 % and 68 %, compared to the 10 to 22 % obtained with traditional biochar production systems. Both biochars exhibited good agronomic properties and fulfilled key quality criteria for soil carbon sequestration, as described by the European Biochar Certificate (EBC) and the International Biochar Initiative (IBI); that is: Organic carbon (Corg) > 50 %, H/Corg < 0.7, and potential toxic elements far below environmental threshold values. Both biochars passed seed germination and worm inhibition toxicity tests. They were characterized by similar physical properties, apart from greater water absorption observed for corncob biochar (CCB). Chemical properties differed; CCB had higher buffer capacity, and was richer in soluble potassium (5473 ppm) and in graphitic like carbon (37.7 %) compared to eucalyptus bark biochar (EB) (897 ppm and 24.9 %). The latter was less alkaline (pH 8.1 versus 9.3 for CCB), had more than twice its electrical conductivity (0.68 dS.m<sup>-1</sup> versus 0.25 dS.m<sup>-1</sup> for CCB) and ash content (10.1 % versus 5.3 %); EB had higher sum of cations. Our results demonstrated that the constructed pyrolyser could be a current and feasible alternative to be used by farmers in Cameroon, to produce a quality biochar product from common residues.

## **Keywords**

Retort kiln, pyrolysis, biochar, physical properties, chemical properties, forest residues, agricultural residues.

## **Résumé**

Dans de nombreux pays en voie de développement, la production de biochar se fait encore principalement avec les fours traditionnels. La présente étude a porté sur la production et la caractérisation de deux biochars à partir des résidus agricoles et forestiers ordinaires au Cameroun : les rafles de maïs et les écorces d'eucalyptus. Pour se faire, nous avons construit et testé Un prototype de four de type « retort » à partir des matériaux locaux. Les rendements obtenus avec ce four variaient entre 33 et 68 %, comparé aux 10 à 22 % obtenus avec les fours traditionnels de production de biochar. Les deux biochars produits présentaient de bonnes propriétés agronomiques et remplissaient les principaux critères de qualité pour la séquestration du carbone dans le sol, comme décrit le « European Biochar Certificate (EBC) » et l'« International Biochar Initiative (IBI) ». Le carbone organique (Corg) > 50 %,  $H / Corg < 0,7$ , et les éléments toxiques potentiels bien inférieurs aux seuils environnementaux. Les deux biochars ont réussi les tests de germination des semences et d'inhibition des vers. Ils ont été caractérisés par des propriétés physiques similaires cependant, on a noté une plus grande absorption d'eau chez le biochar de maïs (CCB). Par contre, les propriétés chimiques des biochars étaient différentes. Le CCB avait une capacité tampon plus élevée et était plus riche en potassium soluble (5473 ppm) et en carbone graphitique (37,7 %) par rapport au biochar de l'écorce d'eucalyptus (EB) (897 ppm et 24,9 %). Ce dernier était moins alcalin (pH 8,1 contre 9,3 pour CCB), avait plus du double de sa conductivité électrique (0,68 dS.m<sup>-1</sup> contre 0,25 dS.m<sup>-1</sup> pour CCB) et de la teneur en cendres (10,1 % contre 5,3 %) ; EB avait une somme plus élevée de cations. Ces résultats ont démontré que le pyrolyseur construit pourrait être une alternative actuelle acceptable à utiliser par les agriculteurs au Cameroun, pour produire un produit biochar de qualité à partir de résidus ordinaires.

## **Mots clés**

Four de type « retort », Pyrolyse, Propriétés physiques, résidus forestiers, résidus agricoles.

## 1. Introduction

Biochar is a product of thermochemical degradation of organic matter under oxygen-deprived conditions. This process, called pyrolysis, can contribute to waste management by using residual biomass (currently improperly managed or unsuitable for conventional discharge) as feedstock. Apart from the solid biochar fraction, pyrolysis also generates gas and liquid fractions that are potential sources of energy (Neves et al. 2011). Biochar production has received increasing attention because of its potential as a soil amendment for soil carbon sequestration and to improve soil quality (Lehmann & Joseph 2009). The potential for biochar to improve soil fertility could increase crop yield on previously degraded soils of smallholder farmers, and consequently, increased food production capacity. This could decrease the need to deforest land for agriculture; deforestation representing a major contributor to greenhouse gases in the atmosphere (Scholz et al. 2014). Simultaneously, biochar offers an alternative for waste management and energy production. Biochar systems are thus particularly relevant in developing countries and could be leveraged to address global challenges associated with food security and climate change mitigation. However, pyrolysis applications are still in their infancy in developing countries and further research and development is needed to fill gaps in knowledge for biochar systems applicable for agricultural production by small landowners.

Biochar characteristics are highly dependent on the feedstock type and pyrolysis conditions (Antal & Grønli 2003; Brownsort 2009; Song & Guo 2012; Crombie et al. 2013). Given the wide range of available raw products and the different production techniques, high variability is to be expected in the physicochemical properties of biochars, and ultimately, in their performance as soil amendments or for soil carbon sequestration (Antal & Grønli 2003). Consequently, the challenge for biochar science is to predict and assure product quality, agronomic benefits and environmental effects of a specific biochar, produced from a given feedstock, under a specific pyrolysis technology and process conditions (Schmidt et al. 2000; Warnock et al. 2007; Lehmann et al. 2011; IBI 2012; Mašek et al. 2013). We therefore need to document properties of raw material, production details and provide a complete characterization of the biochar from these African residues, with reference to standardized definitions set by the European Biochar Certificate (EBC) and the International Biochar

Initiative (IBI). Biochar may be produced with various technologies suitable for either household, medium size or large industrial scales. However, advanced technologies remain beyond reach for rural tropical areas; traditional kiln technologies that do not recycle pyrolysis gases, such as used in Cameroon, dominate in developing countries. This traditional charcoal industry is considered to be both inefficient and polluting, emitting harmful off-gases containing methane, carbon monoxide and particulates (Sparrevik et al. 2014). Retort kilns, which recirculate and combust the pyrolysis gases internally may overcome this problem (Sparrevik et al. 2014).

In developing countries, the progress of research relative to biochar and pyrolysis systems has been restricted, partly by a shortage of facilities for controlled slow pyrolysis of biomass at a scale suitable for agronomic field trials. Our objectives were to produce biochar using a locally constructed retort kiln, then to characterize the biochars in terms of suitability for agronomic use (corn production) or for soil carbon sequestration. We hypothesized that: i) biochar yield with the constructed pyrolyser will be greater than with traditional methods ii) the produced biochar will have different physico-chemical characteristics iii) and will satisfy the norms set by IBI and EBC for biochar definition. Common forestry and agricultural residues in Cameroon were used as raw material: corncob from corn production farm (CCB) and eucalyptus tree bark (EB) from electric poles. Both biochars were then compared, based on standard assays for safe application to soil; our study focuses on production and characterization of these two biochars.

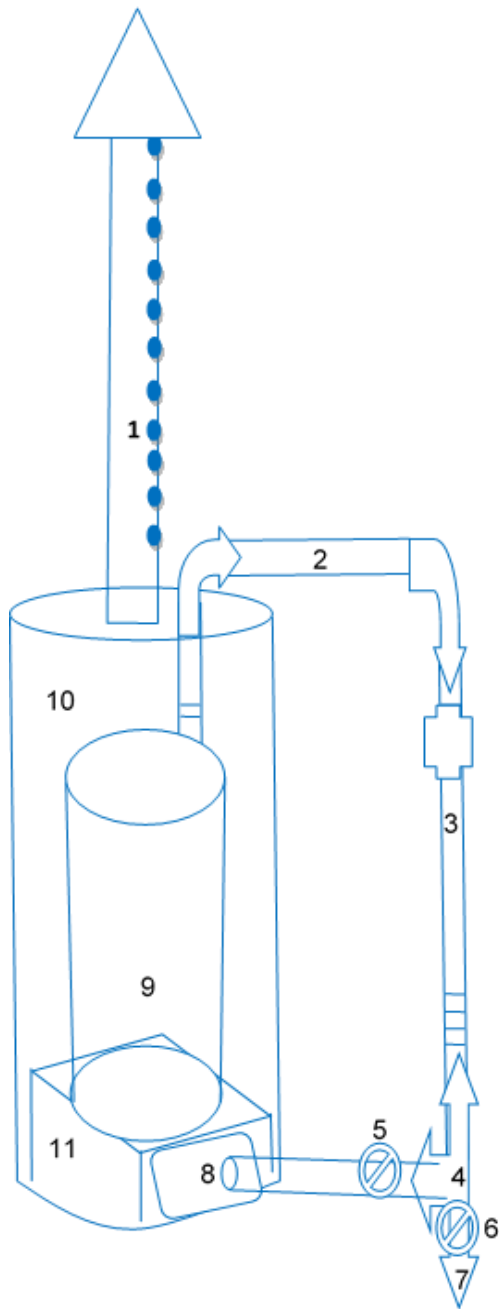
## **2. Material and methods**

### **2.1 *Pilot kiln for pyrolysis***

The pilot pyrolysis plant (Figure 1) is a vertical batch reactor type, constructed using material entirely collected from the Cameroon local market. It includes five main parts: 1) an outer vessel; 2) an inner vessel; 3) gas circulator pipes; 4) a stand for the small vessel; and 5) a chimney. It is designed to operate as batch feeding reactor. The outer vessel is 1.2 m high and 80 cm diameter, while the inner vessel was 90 cm high and 50 cm diameter, equipped with a movable top lid, all made of iron (Figure 1). The chimney was made from a 1.5 mm thin aluminum metal sheet and the stand was constructed with an iron angled bars. This particular size of unit was chosen based on availability of material in the local market and

keeping in mind the ease of transportation and the need for a household scale kiln. Corn was manually harvested from a medium scale (10 ha) corn production farm in Koupara, West Region of Cameroon (5° 30' 0" North, 10° 38' 0" East), then mechanically shelled and sorted.

Collected corncobs had a cylinder-like shape of 3-5 cm diameter and 10 – 20 cm long. Eucalyptus tree bark was manually collected using a cutlass, from an electric pole production site in Bafounda, West Region of Cameroon (5° 34' 40.1664" North; 10° 17' 53.142" East); it had a rectangular shape (10-40 cm long and 5 to 10 mm thick).



**Legend**

- 1 – Chimney
- 2 - Re-circulating piping system
- 3 - Threaded connector
- 4 - T-junction
- 5 – Syngas control valve
- 6 – Pyrolytic liquids control valve
- 7 – Pyrolytic liquids outlet
- 8 – Wood and syngas feed
- 9 – Small vessel (feed of raw material, air tight)
- 10 – Big vessel
- 11 – Stand for small vessel (burning chamber)

**Figure 1. Structure of the pyrolyser**

These organic residues were chosen because of their abundance and their poor economic valorization. Raw materials were sun-dried for about four days after their collection, then weighed and inserted into the small vessel, one type of residue at a time. The top lid of this vessel was then closed for air tightness. The small vessel was placed on top of its stand and the large vessel placed from top as a second ring, making sure the openings for gas recycling were aligned; this allowed the fixation of the gas recycling pipe (Figure 1). Based on 65 production cycles, the recorded complete burning cycle average time was between 5 and 6 hrs. The kiln was first heated for 2 to 3 hours using dry wood (any available hardwood was used). Temperature was recorded using a K-type thermocouple (Model 800024, Geneq Inc), at 3 hrs 45 min, 4 hrs and 4 hrs 15 min, after the beginning of the pyrolysis process. Two K-type probe sensors were introduced inside the large vessel from two holes of 2 mm diameter made side by side on the top. When not in use, the holes were closed with a flat end nail. The kiln was then allowed to cool down for 2 to 3 hours, the system dismantled (gas pipe, then big vessel) and then biochar inside the small vessel was removed and weighed before initiation of a new burning cycle. Biochar yield was calculated using equation 1.

$$B_y(\%) = (B_w/R_w) * 100 \quad (1)$$

Where:  $B_y$  = yield of biochar (% on a mass basis);  $R_w$  = weight of raw dry material (kg);  $B_w$  = weight of biochar (kg).

## 2.2 *Characterization of biochar*

Three composite samples from a mixture of 65 pyrolysis trials of each type of ground biochar, were collected for analysis. Most of the measured parameters were analyzed according to standards established either by the European Biochar Certificate (EBC), the International Biochar Initiative (IBI), or the American Standard Test Methods (ASTM). We included supplementary analyses based on specific environmental safety laws and agricultural use. Each tested property was measured on EB and CCB.

### 2.2.1 *Physical properties*

Tapped bulk density ( $\rho_a$ , g.cm<sup>-3</sup>) was measured using the modified International Standard Organization Method (ASTM 2015). Particle density ( $\rho_s$ , g.cm<sup>-3</sup>) was determined using a gas

displacement pycnometer (AccuPyc 1330 Micromeritics Norcross, GA, USA) (ASTM 2015). The total porosity ( $\Theta$ ) was determined based on the above two parameters using Equation 2 (Flint & Flint 2002).

$$\theta = 1 - \left( \frac{\rho_a}{\rho_s} \right) \quad (2)$$

Particle size distribution was measured using a Tyler sieve instrument (RX-29, Ro-Tap, W.S. Tyler, Mentor, Ohio, USA) and an Allen Bradley sonic sifter based on a two mechanical sieving technique (ASTM 2015). The mean particle diameter (MPD, mm) and uniformity coefficient (UC, %) were calculated using equations 3, 4, and 5 (ASTM 2010)

$$R_i = (F_i/S) * 100 \quad (3)$$

$$MPD (mm) = \frac{\sum_{i=1}^{i=n} (R_i \times N_i)}{100} \quad (4)$$

$$UC (\%) = (D_{60}/D_{10}) * 100 \quad (5)$$

Where:  $F_i$  = fraction weight of sieve number  $i$ ,  $S$  = sum of sieve fraction weights,  $R_i$  = percent retained on sieve number  $i$ ,  $N_i$  = factor for sieve number  $i$  fraction,  $D_i$  expresses the percentage of particle size finer than  $i$ . Biochar capillary rise characteristics were measured by recording the mass of biochar samples initially dry, and then every three days under the following tensions (-140 cm, -100 cm, -75 cm, -50 cm, -25 cm et -5 cm) as in Allaire et al. (2015). Sorption of air moisture by biochar was determined using a sample of 5 g of dry biochar, exposed to air humidity of 80 % in a growth chamber at 22 °C during 72 hrs (Allaire & Parent 2004). Moisture was determined using mass lost during 14 hrs at 105 °C and ash content during 16 hrs at 800 °C in a muffle furnace as adapted from ASTM (ASTM 1990) and CAEAQ (Centre d'expertise en analyse environnementale du Québec et ministere de l'agriculture 2003)..

### 2.2.2 Chemical properties

Volatile matter determination was carried out using the mass lost during 10 min at 950 °C in a muffle furnace (ASTM 1990). Total content of carbonaceous material was calculated by the difference between volatile matter and ash (ASTM 1990). Total carbon ( $C_{tot}$ ) was determined using the standard operating procedure for analysis of total organic carbon in

sediments (dry combustion, IR Detection) as suggested by IBI (US EPA 2005). Graphitic like carbon ( $C_{\text{graph}}$ ) was measured in a LECO elemental analyzer at 1380°C after pre-treating one sub sample with nitric acid.  $C_{\text{org}}$  and  $C_{\text{graph}}$  were measured after pre-treating another sub sample with hydrochloric acid and removal of  $C_{\text{inorg}}$  by volatilization at 1380 °C in a LECO elemental analyzer. The  $C_{\text{org}}$  was calculated as the difference between the  $C_{\text{graph}}$  plus  $C_{\text{org}}$  and  $C_{\text{graph}}$ ;  $C_{\text{inorg}}$  was then calculated as the difference between the  $C_{\text{tot}}$  and the  $C_{\text{org}}$  plus the  $C_{\text{graph}}$  (ASTM 2002; ISO 9686:2 2006; CEAEQ 2015). The pH ( $H_2O$ ) and the electric conductivity (EC) were determined following procedures described by Rajkovich et al (Rajkovich et al. 2011). The pH and EC were then measured using a VWR Symphony SP80PC pH meter, and a Radiometer Copenhagen CDM3 Conductivity Meter, respectively. The buffer capacity consisted in the determination of the volume of HCl (meq) necessary to bring down the biochar pH to either pH=7 or pH=4 (Rajkovich et al. 2011). The solution was prepared as for pH and titrated with 0.5 N HCl using a 140 Corning - pH meter.

Metals and other components (As, Cd, Co, Cr, Cu, Hg, Mo, Ni, P, Pb, Se, Zn); water soluble elements (Ca<sub>sol</sub>, Fe<sub>sol</sub>, K<sub>sol</sub>, Mg<sub>sol</sub>, Mn<sub>sol</sub>, Na<sub>sol</sub>, Al<sub>sol</sub>, Cu<sub>sol</sub>) and exchangeable bases (K, Ca, Mg, Na) were determined by Optical Emission Spectrophotometry with inductively coupled plasma (ICP-OES Optima 4300DV, Perkin-Elmer instrument) in different extracts. For metals and other elements, extract preparation was done using the wet-digestion and dry-ashing methods for total elemental analysis of biochar as described by Enders and Lehmann (Enders & Lehmann 2012). Soluble elements were extracted by agitating 2 g of biochar mixed with 50 ml deionized water for 1 hr on a G10 GYROTORARY shaker, followed by filtration (AGDEX 533 1988). Exchangeable bases were quantified in  $NH_4Cl$ - $BaCl_2$  extract; 2 g of biochar were saturated with 50 ml of 1 N of  $NH_4Cl$ - $BaCl_2$  and shaken for 15 min at 120  $tr.s.min^{-1}$  on a G10 GYROTORARY shaker (Amacher et al. 1990). The sum of the bases, often referred as the cationic exchange capacity (CEC), was calculated as the sum of K, Ca, Mg, Na. Total C, N, S and H contents, that is (CNSH-total) were determined by dry combustion using an elemental analyzer CNS-LECO Truspect (LECO 2009; Brewer et al. 2012; Meng et al. 2013).



### 2.2.3 Toxicity related properties

The potential toxicity of biochar was based on a germination test and a worm avoidance test (Major 2009; Van Zwieten et al. 2010). The germination index was determined using Buttercrunch lettuce (*Lactuca sativa L.*). Dry biochar and commercial garden soil were mixed at the ratio of 0 % of biochar (control), 10 % (volume to volume) and 50 % (volume to volume). The mixtures were moistened and placed in petri dishes. Twenty seeds of lettuce were placed at the soil surface of each dish and installed in a growth chamber programmed for a day-night regime of 16 - 8 hrs, with temperatures of 22 °C and 15 °C. The number of germinated seeds was recorded after 2, 5, 7 and 9 days. A grain was considered as germinated only when cotyledons were completely out of the tegument. The germination index was expressed as the percentage of the control (CEAEQ 2003; Mala & Babu 2005; Mitelut & Popa 2011). *Eisenia fetida* sp. was used for the worm avoidance test. The same biochars and soil were mixed at 10 % and 50 % (volume to volume). They were moistened and inserted into half of an 8 cm x 18.5 cm x 6 cm box. The other half contained the moistened soil only (0 % biochar), leaving a small clearance in between both, where 20 worms were placed. After 48 hrs, the number of worms in each side of the box were counted and the worm avoidance index was expressed as percentage of the control (CEAEQ 2003; Mala & Babu 2005; Mitelut & Popa 2011).

### 2.3 Statistical analyses

R software v.3.2 for Windows was used for statistical analysis. A Wilcoxon test was used at a 10% significance level to compare mean values of the two biochars. We selected this level of significance because of the low number of replicates.

## 3. Results and Discussion

A summary of all results including a comparison to the criteria set by the IBI and EBC can be found in Table 1. CCB and EB were well within the threshold criterion set by these organizations. Supplementary analyses were completed for a better understanding of implications for agricultural production or impacts on the environment.

### 3.1 *Pilot kiln for pyrolysis*

The construction cost of the retort kiln was estimated to one hundred thousand francs cfa. (163 US\$). This cost is judged affordable for local farmers as a medium term investment based on their annual income. During kiln operation, the average temperature value recorded was  $281.4 \pm 37.6$  °C. This temperature was relatively low compared to other values reported in the literature for pyrolysis. According to Stelt et al. (2011), our process could be classified as torrefaction rather than pyrolysis. The relatively low temperature could be due to poor external insulation of the kiln and we expect to improve the future design to address this problem. The standard variation of 13.4 % in temperature was within the acceptable range of 20 %, as recommended by the EBC (EBC 2012). Within this range, it is assumed that variation in temperature does not significantly influence biochar properties. The average time for pyrolysis was 5 to 6 hours. Production of gases sustaining combustion stopped between 4 and 5 hrs after initiation of pyrolysis, wood was less often used to complete the process. During operation of the kiln, the smoke produced was lighter compared to that observed when using traditional earth-mound kilns. The average yield of biochar varied with the raw material. It was 33 % for CCB and 68 % for the EB. This difference can be partially explained by the difference in density between the two raw materials. These yields were relatively high compared to 30 - 40 % obtained in Kenya using similar technology (Adam 2009) and far better than the 10 to 22 % usually obtained with traditional systems. The initial shape of the raw material was maintained once pyrolysed, grinding was thus necessary for uniform application in the field; this was done using a mill powered with a diesel motor. After grinding, a net color distinction was observed; the CCB was darker than EB, probably due to the level of carbonization of each raw material.

### 3.2 General properties of raw material and changes after pyrolysis

Both raw materials show similar  $C_{tot}$ , H, O,  $O/C_{tot}$ ,  $N_{tot}$ , and water content; only ash content differed (

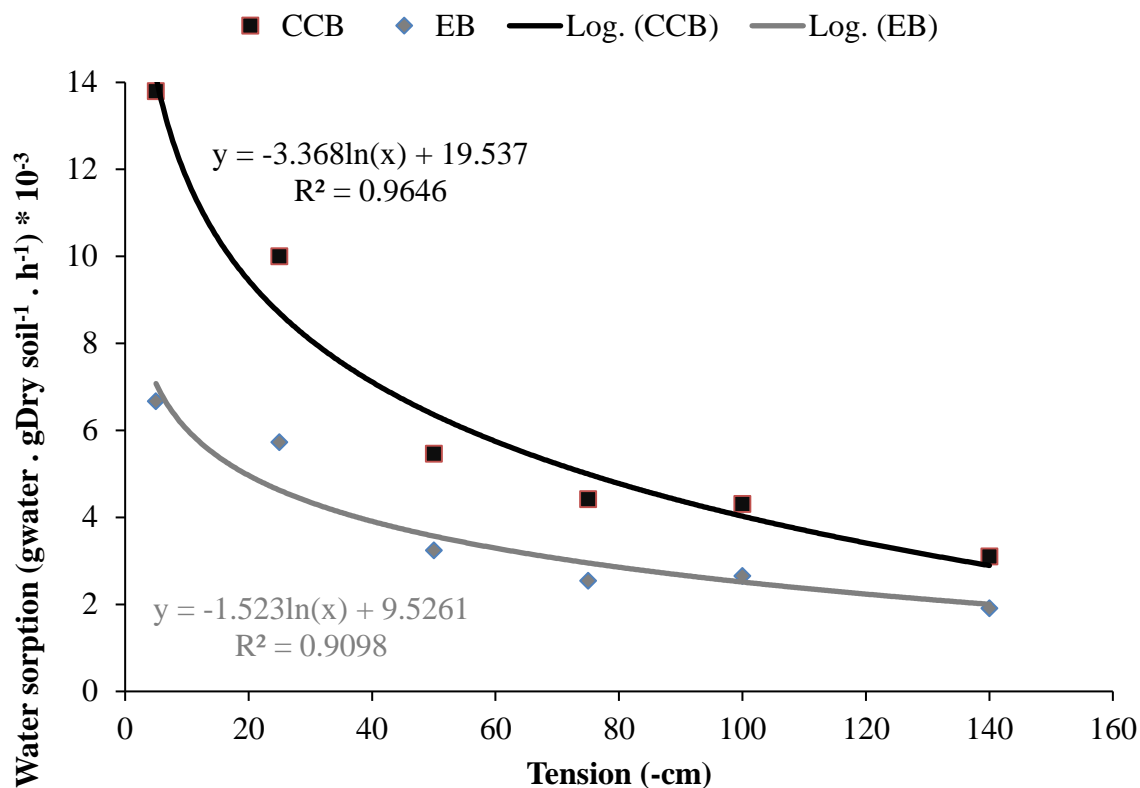


Figure 2). The elemental composition of raw materials ranged from 44.2 to 47.2 % of  $C_{tot}$ ; 40.5 % of oxygen and approximately 5.5 % of hydrogen. These values are similar to those presented in reviews including different types of pyrolysis and raw materials (Neves et al. 2011; Zhao et al. 2013; Allaire et al. 2015).

**Table 1 : Chemical properties of corncob biochar (CCB) and eucalyptus biochar (EB) and comparison with the international biochar initiative (IBI) and the European Biochar (EBC) standards.**

Parameters	Symbols	Units	Corncob biochar	Eucalyptus biochar	EBC Norms	IBI Norms
<b>Biochar physical parameters</b>						
Bulk density	$\rho_a$	$\text{g cm}^{-3}$	0.33	0.46	Not required	Not required
Particle density	$\rho_s$	$\text{g cm}^{-3}$	1.62	1.63	Not required	Not required
Porosity	$\Theta$	$\text{m}^3 \text{m}^{-3}$	0.79	0.72	Not required	Not required
Uniformity coefficient	UC (D60/D10)	%	212	243	Not required	Not required
Mean Particle Diameter	MPD	mm	0.24	0.13	Not required	Not required
Capillary rise	$\Theta_{-0.05 \text{ m}}$	%	13.80	6.70	Not required	Not required
	$\Theta_{-1.4 \text{ m}}$	%	3.10	1.91	Not required	Not required
Air humidity sorption	$\Theta_x$	%	6.17	6.14	Not required	Not required
Moisture content	$\Theta_g$	% dry mass	4.46	5.57	Declaration	Declaration
Specific surface area	SSA	$\text{m}^2 \text{g}^{-1}$	NA	NA	Declaration	Declaration if > 150
<b>Biochar chemical parameters</b>						
Total ash		% dry mass	5.3	10*	Declaration	Declaration
Volatile mater	VM	% dry mass	29.3	26.5	Declaration	Declaration
Total carbon	$C_{\text{tot}}$	% dry mass	68.8	56.2	$C_{\text{tot}} \geq 50$	Not required
Graphitic like carbon	$C_{\text{graph.}}$	% of $C_{\text{tot}}$	54.9	44.4	10-40 % of $C_{\text{tot}}$	Not required
Organic carbon	$C_{\text{org.}}$	% dry mass	29.7	27.8	Not required	Class 1 : $\geq 60$ $\geq 30$ Class 2 < 60 Class 3 : $\geq 10$ < 30 Class 4 : < 10 not a biochar
Inorganic carbon	$C_{\text{inorg.}}$	% dry mass	1.3	3.5	Not required	Not required
Total nitrogen	N	% dry mass	0.9*	0.5	$\leq 0.4$	Declaration

Parameters	Symbols	Units	Corncob biochar	Eucalyptus biochar	EBC Norms	IBI Norms
Hydrogen	H	% dry mass	3.83	3.26	/	/
Oxygen	O	% dry mass	14.7	27.4	/	/
Hydrogen-carbon ratio	H:Corg	/	0.13	0.12	≤ 0.7	≤ 0.7
Total nitrogen		% dry mass	0.9*	0.5	≤ 0.4	Declaration
Total phosphorus	P	mg kg <sup>-1</sup>	1780.4*	1659.1	Declaration	Optional
Electrical conductivity	EC	dS m <sup>-1</sup>	0.28	0.68	Declaration	Declaration
Buffer capacity	pH4	meq HCl	0.64	0.19	Not required	Not required
	pH 7	meq HCl	0.1	0.04	Not required	Not required
Liming power	/	%CaCO <sub>3</sub>	NA	NA	Not required	Declaration if pH >7
Particle size distribution	/	0.05<%<2 mm	97	89	Not required	Declaration
		0.025<%<0.05	3	10	Not required	Declaration
		< 0.025 mm	0	1	Not required	Declaration
Hydrogen potential	pH water	/	9.3	8.1	Declaration if pH > 10	Declaration
<b>Metal and other components</b>						
Cobalt	Co	mg kg <sup>-1</sup>	1.4±0.1	3.5±0.2*	/	40-150
Chromium	Cr	mg kg <sup>-1</sup>	0.2±0.0	1.2±0.1*	< 90	64-1200
Copper	Cu	mg kg <sup>-1</sup>	6.1±0.3	9.5±0.7*	< 100	63-1500
Zinc	Zn	mg kg <sup>-1</sup>	139±5*	18±1.4	< 400	200-7000
Arsenic	As	mg kg <sup>-1</sup>		< 0.0	Not required	12-100
Cadmium	Cd	mg kg <sup>-1</sup>		< 0.0	< 1.5	1.4-39
Mercury	Hg	ng g <sup>-1</sup>		< 0.1	< 1	< 0,061
Molybdenum	Mo	mg kg <sup>-1</sup>		< 0.0	Not required	< 0,0079
Nickel	Ni	mg kg <sup>-1</sup>		< 0.0	< 50	47-600

Parameters	Symbols	Units	Corncob biochar	Eucalyptus biochar	EBC Norms	IBI Norms
Lead	Pb	mg kg <sup>-1</sup>		< 0.0	< 120	70-500
Selenium	Se	mg kg <sup>-1</sup>		< 0.0	Not required	1-36
Chlorine	Cl	mg kg <sup>-1</sup>	NA	NA	Declaration	Not required
Polychlorinated dibenzo-p-dioxins and dibenzofurans	PCDD/Fs	ng kg <sup>-1</sup> I-TEQ	NA	NA	Required	≤ 9
Polycyclic aromatic hydrocarbons	PAHs	mg kg <sup>-1</sup> t	NA	NA	Required	6 - 20
<b>Water soluble elements</b>						
Manganese	Mn_sol.	mg kg <sup>-1</sup>	0.63	12*	Not required	Not required
iron	Fe_sol.	mg kg <sup>-1</sup>	8	2.7*	Not required	Not required
Aluminium	Al_sol.	mg kg <sup>-1</sup>	5	1.4*	Not required	Not required
Copper	Cu_sol.	mg kg <sup>-1</sup>	0.6	0.3	Not required	Not required
Zinc	Zn_sol.	mg kg <sup>-1</sup>	0.52	0.02*	Not required	Not required
Potassium	K_sol.	mg kg <sup>-1</sup>	5473	897*	Not required	Not required
Calcium	Ca_sol.	mg kg <sup>-1</sup>	21	398*	Not required	Not required
Magnesium	Mg_sol.	mg kg <sup>-1</sup>	33	191*	Not required	Not required
Sodium	Na_sol.	mg kg <sup>-1</sup>	94	180*	Not required	Not required
<b>Exchangeable bases</b>						
Calcium	Ca	cmol(+) kg <sup>-1</sup>	0.81	14.73*	Not required	Not required
Potassium	K	cmol(+) kg <sup>-1</sup>	26.47	7.54*		
Sodium	Na	cmol(+) kg <sup>-1</sup>	0.50	0.96*	Declaration	/
Magnesium	Mg	cmol(+) kg <sup>-1</sup>	0.78	1.02	Not required	Not required
Cation exchange capacity	CEC	cmol(+) kg <sup>-1</sup>	28.55	24.24	Not required	Not required

<b>Parameters</b>	<b>Symbols</b>	<b>Units</b>	<b>Corn cob biochar</b>	<b>Eucalyptus biochar</b>	<b>EBC Norms</b>	<b>IBI Norms</b>
<b>Toxicity related properties</b>						
Earthworm avoidance test	Avoidance index	%	1	1.3	Not required	<1: avoidance ≥1: no avoidance
Germination test	Germination index	%	1	1	Not required	<1: fail ≥1: pass

Value followed by the symbol \* is significantly different from its side by side at 10 % level of significance.  
NA = not available.

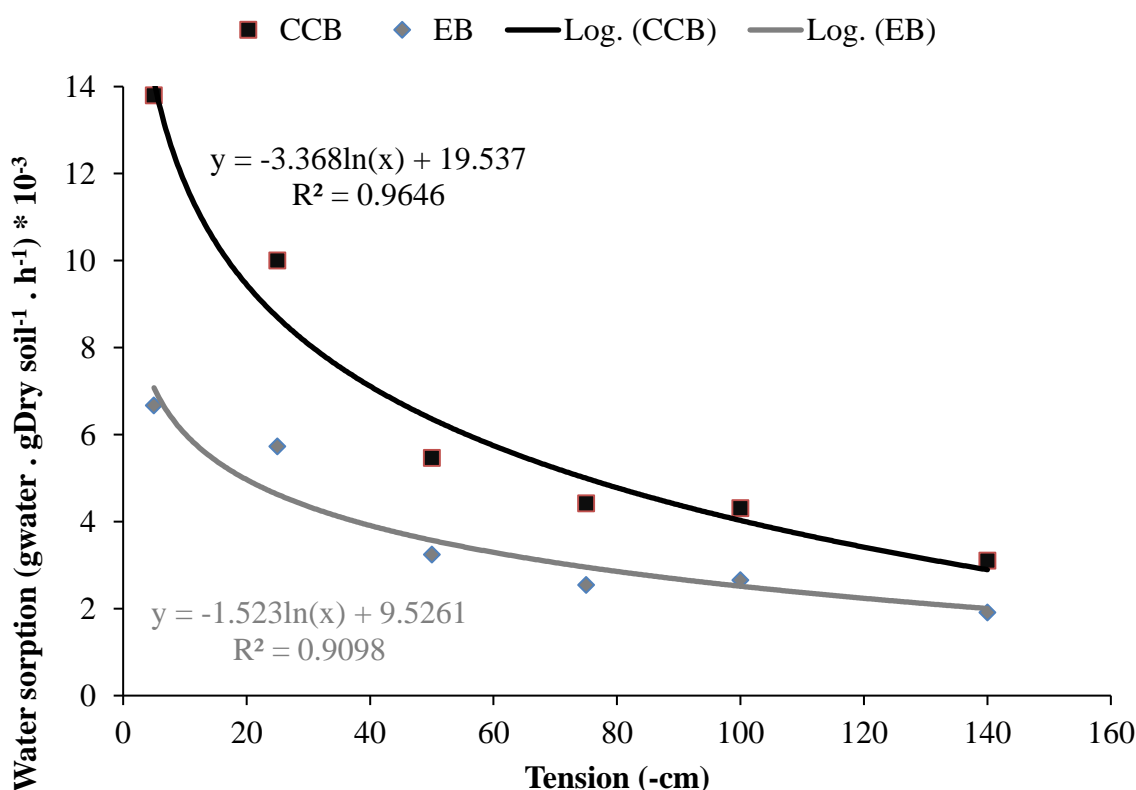
These studies reported intervals of 40 to 60 % for  $C_{tot}$ , 30 to 50 % for oxygen and from 5 to 8 % for hydrogen. Nitrogen (0.4 to 0.7 %) and sulfur (0 %) content were low in our raw materials, indicating that should produce less NO<sub>x</sub> and SO<sub>2</sub> emissions during combustion (Neves et al. 2011). Molar O/ $C_{tot}$  ratio dropped considerably from the biomass state to the coal state, pyrolysis effectively contributed in concentrating more carbon in our biochar; similar results were observed by Ahmad and Subawi (Ahmad & Subawi 2013) describing the Van Krevelen diagram for different materials. Ash content was 1.6 % in CCB and 4.5 % in EB. These values were similar to those described before (Guerrero et al. 2005; Liu et al. 2014). EB will therefore supply more nutrients to soil when used as amendment, potentially improving soil fertility (Zhao et al. 2013). The raw biomass with the highest volatile matter generated biochar with the greatest volatile matter content (Table 1). This tendency was reported in previous studies (Meng et al. 2013; Liu et al. 2014). Pyrolysis reduced volatile matter by 60 % in both feedstock, indicating that gaseous fuel was present in the raw materials, thus predisposing them to easy ignition, even at relatively low temperature (Neves et al. 2011). This could be advantageous for the pyrolysis process when using less energy efficient technology and relatively low temperatures such as in the present study. The combination of high ash content and low volatile matter content lead to a higher biochar yield for EB compared to CCB.

### 3.3 *Physical properties of biochars*

Moisture content was similar between biochars (Figure 2). The initial water content of CCB and EB was around 5 % (Table 1), slightly higher than the average of 40 biochars shown in Allaire et al. (2015). The biochars exhibit different levels of air humidity sorption, also known as hygroscopicity. These biochars were slightly hygroscopic, with an average air humidity sorption value of 6.15 % over 72 hrs under 80 % relative humidity level. Considering that in dry soils, the air relative humidity is generally 100 %, 1 g of this biochar is able to fix up to 0.06 g of water vapour. Similar and much higher hygroscopicity values were reported by Allaire et al. (2015). This property is of utmost importance relative to handling, storage, and agricultural water management, as it helps soil to retain moisture during dry conditions. Field application of dry biochar with this characteristic may be



problematic under windy conditions, because it may have difficulty in rewetting, as observed for peat (Sheiman 1965).

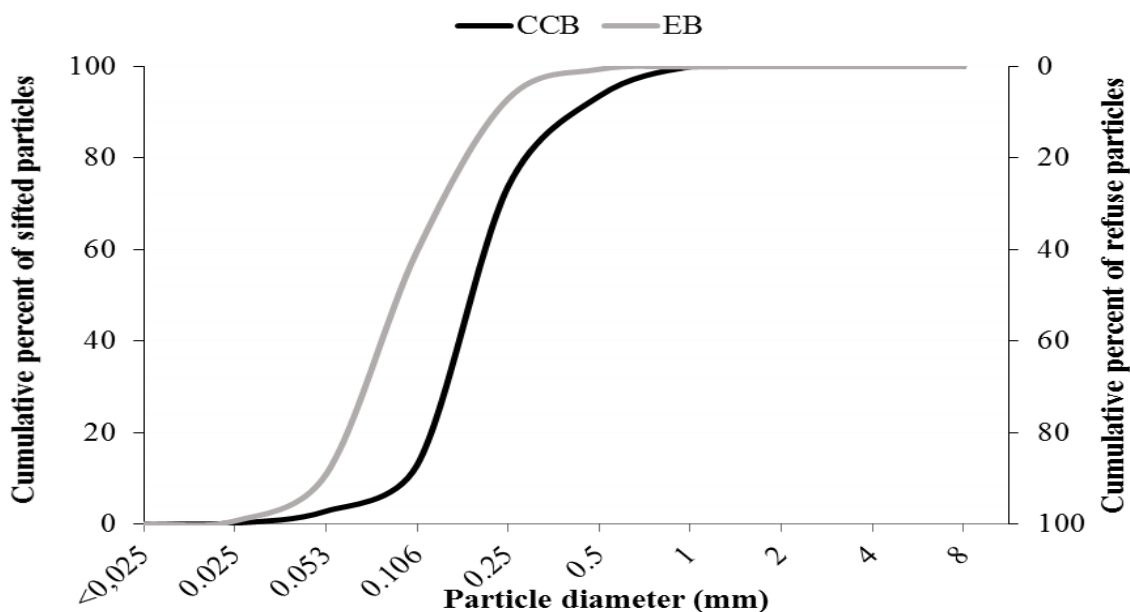


**Figure 2. Biochar water sorption by capillary rise under different tensions for corncob biochar (CCB) and eucalyptus biochar. (EB)**

Water sorption by capillary rise reveals that CCB biochar absorbs twice the amount of water absorbed by EB under the same conditions at low tension (- 0.05 m) and around one and half times more at higher tension (- 1.40 m). Therefore, we expect that soil water retention of soils amended with CCB may be slightly better compared to that for soils amended with EB. Mean particle diameter (MPD) were respectively 0.24 mm for CCB and 0.13 mm for EB. It is an important criterion to be considered for mechanized application, since it influences the ease of flow of the product that may have a tendency to clog the nozzles of application equipment. This is of less concern in Africa, since most agricultural activities are carried out manually, mechanisation being less developed. It also influences water retention. The uniformity coefficient (UC = D60/D10) of both biochars was lower than three (2.12 and 2.43 respectively for CCB and EB). Thus, particle sizes were uniformly distributed and their size distribution fell within the range of sand, in reference to soil texture classification. EB

particles were finer than CCB, 93 % of EB particles were below 0.25 mm versus 74 % for CCB. Also, 67 % of EB particles fell below 0.1 mm comparatively to only 37 % for CCB (Figure 3). Handreck (Handreck 1983), studying the relationship between water and soil particle distribution concluded that generally, increasing proportions of finer material (< 0.1 mm) progressively decrease both air-filled porosity and water release at low tension. We thus expect that CCB will have a greater ability to supply water at low suctions (up to 300 kPa), while EB will potentially release more water at higher suctions (below 10 kPa).

Based on conclusions of study by Verheijen et al. (2010), the addition of biochar with much finer particle size (< 0.1 mm) as EB to soil may also improve sorption of common environmental pollutants. Both biochars had relatively high porosity, 80 % for CCB and 72 % for EB. Once incorporated to soil, they could thus contribute to good air flow and provide potential habitat for microorganisms (Lehmann et al. 2011). Tapped bulk density (after grinding), as requested by EBC, was low for these biochars (0.33 g cm<sup>-3</sup> for CCB and 0.46 g cm<sup>-3</sup> for EB); these values are similar to those reported in the literature by Allaire et al. (2015). Bulk density values fell within the ideal range of 0.3 - 0.6 g cm<sup>-3</sup>, as suggested by Kaudal et al. (2015) and seem not to have been influenced by feedstock type, perhaps explained by the physical grinding of both biochars after pyrolysis.



**Figure 3. Particle size distribution of corncob biochar (CCB) and eucalyptus biochar (EB)**

Average particle density ( $\rho_s$ ) of both biochars was  $1.62 \text{ g.cm}^{-3}$ , which is similar to that of organic soil (Redding & Devito 2006). We thus expect that when applied to local soils, for which bulk density ranges from  $0.8$  to  $1.2 \text{ g cm}^{-3}$ , the biochar could contribute to reduce soil bulk density. High porosity and low bulk density of biochar could contribute to improved soil aeration and could positively impact soil water availability. Based on this, both biochars could be recommended for agricultural use.

### 3.4 *Chemical properties of biochars*

The  $C_{\text{tot}}$  of both biochars was greater than 50 %, as recommended by the EBC (EBC & IBI 2014). The IBI use the  $C_{\text{org}}$  content as criteria for biochar classification (EBC & IBI 2014). According to this criteria, CCB and EB fell in class 3 ( $>10 \% C_{\text{org}} < 30 \%$ ), (Table 1.). The CCB and EC biochars exhibit low  $H/C_{\text{tot}}$  and  $O/C_{\text{tot}}$  molar ratios (Table 1). A recent study (Spokas 2010) suggested that such biochars with molar  $O/C_{\text{tot}}$  ratio in the range of  $0.2 - 0.6$  have intermediate half-lives in the soil between 10 to 1000 years. CCB and EB, with molar  $O/C$  of  $0.21$  for CCB and  $0.49$  for EB, are expected to be more stable in soil compared to their equivalent raw biomass (molar  $O/C$  of  $0.86$  for corncob and  $0.92$  for eucalyptus bark), enabling them to act as a carbon sink (Enders et al. 2012). The criteria of  $H/C_{\text{org}} < 0.7 \%$  set by EBC and IBI (Table 1.) was also met by both biochars. The  $C_{\text{graph}}$  value was 29 % for CCB and 27 % for EB. This carbon fraction is the most stable with time and generally increases with pyrolysis temperature and degree of lignification of the raw material (Krull 2010; Singh et al. 2012). Allaire et al. (2015) reported values for  $C_{\text{graph}}$  ranging from 72 % for maple tree wood, to 10 % for agricultural residues (leek). Apart from ash and  $C_{\text{inorg}}$ , all remaining parameters are slightly higher for CCB compared to EB.

Inorganic carbon, also referred as carbonate carbon (Wang et al. 2014) is not mentioned by either regulatory board as an important property for biochar quality definition, but it may be important for agriculture, as it may contribute to improve the liming potential of biochars (Wang et al. 2014). Graphitic like carbon, also not mentioned by IBI or EBC, provides an indication of the importance of the most refractory fraction (most stable portion) of carbon in biochar. Based on described parameters and on comparative information in Table 1, CCB is apparently more aromatic and will potentially be more stable in the soil compared to EB.

Total nitrogen for EB (0.47 %) was slightly above the minimum threshold of 0.4 % fixed by the EBC. The level for CCB (0.88 %) was two times higher. This can be an advantage when applied to infertile soils; IBI only requests a declaration of nitrogen content (without indicating a threshold). The C/N ratio was 78 for CCB and 120 for EB; these high C/N ratios could lead to soil nitrogen immobilization, rendering it unavailable for plants for a certain period.

Both biochars were basic (Table 1). The CCB was at pH 9.3 and EB at 8.1. The pH of the amendment is critical for soil nutrition and crop productivity. Soil pH values of 6 to 7 are considered optimal for growth of most crops (Lee et al. 2013). Acid soils may be improved by adding materials that increase their pH, such as lime or biochars that are basic. However, application of such alkaline biochars is not suitable for alkaline soils. The buffer capacity at pH4 was 0.64 meq HCl for CCB and 0.19 for EB and at pH7 was 0.1 for CCB and 0.04 for EB. CCB might better stabilize soil pH for improving plant growth compared to EB. The CCB buffering capacity is similar to that of wood biochars, while EB is similar to that of biochars made from non-ligneous materials (Allaire et al. 2015). The electrical conductivity values, 0.28 dS.m<sup>-1</sup> for CCB and 0.68 dS m<sup>-1</sup> for EB, reflected the amount of total dissolved salts in the biochar. The high value of electrical conductivity in EB compared to CCB is related to its high ash content. Many plants experience stress due to salts, when the soil electrical conductivity exceeds 0.25 dS m<sup>-1</sup> or 2.0 dS m<sup>-1</sup> (Hanlon 2012). Given the dilution effect in the soil, the values were acceptable for application to agronomic soil.

Except for potassium, other soluble elements are not requested as standards by EBC and IBI. However, the evaluation of sum of bases (CEC) is recommended. Exchangeable K was higher for CCB compared to EB, while exchangeable Ca was higher for EB. High Ca levels have been reported for red oak and yellow-poplar biochar (Jin et al. 2013). Other studies (Mukherjee et al. 2011; Budai et al. 2014) revealed that the sum of bases of biochar was temperature dependent, with biochar produced at lower temperature, as for CCB and EB, having higher values. The values of 24 and 29 cmol(+).kg<sup>-1</sup> obtained respectively for CCB and EB fall within the mid-range of 3-69 cmol(+).kg<sup>-1</sup>, as reported by other researchers (Mukome et al. 2013; Mitchell et al. 2013). Sodium and Mg contributed less to the sum of bases, which is high compared to that of mineral soil (15 cmol(+) kg<sup>-1</sup>), but low compared to

values  $> 100 \text{ cmol}(+) \text{ kg}^{-1}$  observed for humic substances, montmorillonite and vermiculite (Sohi et al. 2010; Mitchell et al. 2013).

Total phosphorus content of CCB was 0.18 % and 0.17 % for EB, These values are relatively high compared to tropical soil average values presented by Yerima K. & Van Ranst (2005). Low values of 0.12 % were observed by the latter author in ferrallitic soils of Cameroon. Phosphorus is an essential plant nutrient for physiological processes related to energy utilization, and thus CCB and EB with their level of total phosphorus may act as a fertilizer. However, biochars with high available phosphorus could present a concern in terms of surface water contamination (Denyes et al. 2013). Both biochars were potential sources of phosphorus to soils and risks related to their application will vary accordingly to its solubility and soil properties.

Water solubility of the exchangeable elements in biochar was assessed, though not requested by the regulatory boards. Except for copper, all the measured elements (Ca\_sol, Fe\_sol, K\_sol, Mg\_sol, Mn\_sol, Na\_sol, Al\_sol, Cu\_sol, Zn) differed between the two biochars at a significance level of 10%. Soluble elements give an indication of nutrients that are easily available to plants when biochar enters the soil solution and also helps to evaluate potential environmental pollution risks (leaching) associated with biochar use in agricultural soil. Our results indicate that the EB will easily liberate more Na, Mg, Ca, Fe and Mn, once in contact with water, while CCB will liberate more K, Fe, Al, Cu and Zn. High iron and Al toxicity, as well as Ca and Mg deficiency are often reported in oxisols, where this biochar will be applied (Yerima K. & Van Ranst 2005). Both biochars have low Al and Fe values and relatively high Ca and Mg values, which is appropriate for these soils. Soluble K values are above those generally encountered in oxisols (Yerima K. & Van Ranst 2005) but its excess in soils is not generally considered an agronomic problem.

### 3.5 *Toxicity related properties*

Biological testing of biochar is important to assess its toxicity to soil invertebrates and plants. The germination assays and earthworm avoidance test demonstrate that there is neither inhibition of germination, nor preference in the repartition of worms according to biochar type, at 10 % volume by volume. This was best reflected by the germination index of 1.0

after 10 days, for both biochars, and worm avoidance index after 48 hours of 0.9 and 1.3 respectively for CCB and EB. At a higher level of 50 % volume by volume, EB tends to slow germination rate (germination index of 0.65), while worms prefer this mixture compared to normal soil (worm avoidance index of 13.3). These results corroborate those obtained with poultry, corn stover, eucalyptus and fresh pine biochar by other researchers (Chan et al. 2008; Free et al. 2010; Van Zwieten et al. 2010). Reduced and slow germination rate with higher biochar concentration as observed in this study were previously described by (Allaire et al. 2015). These authors also noticed that worms preferred soils with 50 % biochar from hardwood but totally avoided soil mixed with activated biochar, and were indifferent for the same concentration of biochar from other feedstocks. One explanation to the decrease in germination rate could be reduced initial moisture due to biochar hydrophobicity; reduced worm avoidance could be due to the recalcitrant nature of biochar. Our biochars can thus be safely applied to soil as amendment. Heavy metals, metalloids and other potential toxic elements values as reported in (Table 1) are far below thresholds.

Based on all the above properties in relation to relevant indicators and thresholds defined by IBI and EBC, we are able to recommend unrestricted application of both biochars to agricultural soils and their application would seem particularly pertinent to oxisol soils of the study region.

#### **4. Conclusion**

The retort kiln was effectively constructed with local material and is affordable for the local population. During its operation, syngas was recycled to maintain the pyrolysis process and the produced smoke was lighter compared to that observed with traditional kilns. The resulting biochars fulfilled all the basic criteria set out by EBC and IBI for biochar definition and are acceptable as class 3 biochar according to IBI. Further analysis of both biochars revealed appreciable characteristics that could be of importance to agricultural production and management in highly weathered soils, in the context of climate change and water deficiencies. Some of these include high pH and good buffer capacity, high content of P and soluble Ca, Mg and K and good water retention potential. Both biochars exhibited good agronomic properties and relatively similar physical properties, with water absorption capacity of CCB being twice that of EB and high porosity levels for both biochars. Chemical

properties differed; apart from a high soluble K content, we observed that EB has higher soluble elements and nitrogen content compared to CCB; the latter has a relatively higher carbon content in all forms, mainly graphitic, compared to EB. We thus expect better short term positive effects of EB for agricultural production, while CCB could generate longer term effects for soil carbon sequestration, due to its more recalcitrant nature. In the next research and application steps, we plan to improve the insulation of our retort kiln for better energy efficiency. However, the present results can already be used to stimulate cleaner biochar production for agricultural use in developing countries, with potential multiplicative positive effects for local farmers.

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## **Chapitre II**

### **Quantifying the influence of Eucalyptus bark and corncob biochars on the physico-chemical properties of a tropical oxisol under two soil tillage modes<sup>2</sup>**

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<sup>2</sup> Article soumis sous peu: Djousse, K.B.M., Allaire, S.E., Munson, A.D., Quantifying the influence of eucalyptus bark and corncob biochars on the physico-chemical properties of a tropical oxisol under two soil tillage modes.

## **Abstract**

This study aimed to assess the impact of two biochars applied at the rate of 15 t ha<sup>-1</sup> on physico-chemical parameters of an oxisol in Cameroon. The biochars were made from slow pyrolysis (~300 °C, 4 hrs) of eucalyptus tree bark and corncobs and then incorporated into the top 15 cm of the soil. A split plot design was used with soil tillage mode (flat plots (FP) or furrows and ridges (FR)) in the main plot and five treatments on subplots. Soil porosity, bulk density, saturated hydraulic conductivity, available water content, pH, N, P, K, CEC, and EC were analysed before biochar application, then 6 and 12 months after. None of the measured soil physical parameters were affected by the presence or type of biochar. The total porosity was lower during the second production period compared to the first, while available water content and van Genuchten parameters increased during the second production period. No significant difference was observed between soil N, P, K, Na, CEC and EC of control and treated plots. These results could be explained by the low rate of biochar addition into a soil with good structure and water retention, and the short duration of the experiment. However, soil pH and organic carbon significantly increased in all treated plots compared to control.

## **Keywords**

Tropical oxisol, physical parameters, chemical parameters, biochar, furrows and ridges.

## **Résumé**

Cette étude évalue l'effet de deux biochars appliqués au taux de  $15 \text{ t ha}^{-1}$ , sur les propriétés physico-chimiques d'un oxisol au Cameroun. Les biochars utilisés ont été fabriqués à base d'écorces d'eucalyptus et de rafles de maïs sous pyrolyse lente ( $\sim 300 \text{ }^\circ\text{C}$ , 4 heures) ; puis incorporés dans les 15 premiers cm du sol. Un dispositif en split-plot a été mis sur pieds avec en parcelles principales le mode de travail du sol qui était soit le labour à plat (FP) ou le labour en sillons-billons (FR) et en sous parcelle les traitements. La porosité du sol, sa densité apparente, sa conductivité hydraulique saturée, sa teneur en eau disponible, le pH, le Corg, la teneur en N, P, K, la CEC et la EC ont été déterminés avant l'application du biochar, puis 6 et 12 mois après. Aucun des paramètres physiques mesurés du sol n'a été affecté statistiquement par la présence ou le type de biochar. La porosité totale a été plus faible pendant la deuxième période de production relativement à la première, alors que la teneur en eau disponible et les paramètres Van Genuchten ont augmenté au cours de la deuxième période de production. Aucune différence significative n'a été observée entre les parcelles traitées au biochar et les parcelles témoins, pour ce qui concerne la teneur du sol en N, P, K, Na, leur CEC et leur EC. Ces résultats s'expliquent par le faible taux d'addition de biochar dans un sol avec une bonne structure et une bonne rétention d'eau ainsi que la courte durée de l'expérience. Cependant, le pH du sol et le carbone organique ont considérablement augmenté dans toutes les parcelles traitées par rapport à la parcelle témoin.

## **Mots clés**

Oxisols tropicaux, paramètres physiques, paramètres chimiques, sillon et billons.

## 1. Introduction

Biochar is the porous carbonaceous solid produced by pyrolysis i.e. thermochemical conversion of organic materials in an oxygen depleted atmosphere. Its physico-chemical properties have potential to contribute to long-term storage of carbon in the soil and improvement of soil structure and fertility (Demirbas & Arin 2002). Biochars made from diverse biomass are characterized by different morphological and physico-chemical properties, and also differ based on pyrolysis conditions, including temperature, rate and duration (Mukherjee et al. 2011; Butnan et al. 2015). Biochar has the potential to improve fertility of degraded soils either by direct supply of nutrients, by fixing nutrients followed by subsequent slow release or by improving soil structure and water retention (Unger et al. 2011). In tropical areas such as in Cameroon, oxisols are among the dominant soil types. They are characterized by an acidic pH (3 - 5.5), high concentration of heavy metals, Al and Fe toxicities and low cation exchange capacity (CEC), all which limit plant nutrient availability, resulting in lower yield (Chintala et al. 2012). Organic and inorganic fertilizers could contribute to maintain or increase the fertility of these soils. However, under the economic conditions prevailing in many sub-Saharan African countries, resource-poor farmers use little chemical fertiliser (Craswell & Vlek 2013). In addition, benefits only last for a few growing seasons, since added nutrients are prone to leaching, given the low CEC of oxisols (5-15 cmol kg<sup>-1</sup>) (Baligar & Bennett 1986). In these acidic soils, biochar has been shown to improve the holding capacity of nutrients, including: phosphorus (P), calcium (Ca), potassium (K), magnesium (Mg), sulfur (S), and nitrogen (N) (Mann 2002). Improvement of soil pH, electrical conductivity (EC), CEC and soil C were also reported (Chintala et al. 2014; Sohi et al. 2009).

### 1.1 *Biochar and soil physical properties*

Soil physical properties largely determine rooting depth and the availability of air and water within the rooting zone (Downie 2009). Bulk density ( $\rho_a$ ) is one of the most important soil characteristics affecting rainfall infiltration (Ueckert et al., 1978). In a meta-analysis, Omondi et al. (2016) obtained an average value of 7.6 % reduction in  $\rho_a$  following biochar application; this was attributed to the initial low  $\rho_a$  of biochar. Biochar impact on soil  $\rho_a$  varies, however, with application rate and soil type. Biochar amendment at 10 t ha<sup>-1</sup>

significantly reduced soil  $\rho_a$  in an Alfisol low in organic carbon, but had no effect in an Andosol high in organic carbon (Herath et al. 2013). Ventura et al. (2013) revealed an inverse linear correlation between  $\rho_a$  and biochar application rates (30 and 60 t ha<sup>-1</sup>) for 5 cm and 10 cm depths, on a sub-alkaline clay loam soil. Soil total porosity ( $\Theta$ ) affects rooting zone processes such as plant water uptake and soil microbial respiration by influencing gas movement (Hillel 2004). Increase in soil  $\Theta$  after biochar application was found to be rate and soil specific: 4 %, 3.5 %, 8.6 % and 19 % increases were recorded respectively for low (<20 t ha<sup>-1</sup>), medium (21– 40 t ha<sup>-1</sup>), high (41–80 t ha<sup>-1</sup>) and very high (>80 t ha<sup>-1</sup>) application rates (Omondi et al. 2016). The same author also noted an increase of 7.5 % in  $\Theta$  in coarse textured soils (sandy loam and coarser) and 7.1 % in fine textured soils (clay loam and finer). However, Hardie et al. (2013) found no influence of biochar on  $\Theta$  by either direct pore contribution, creation of accommodation pores, or improved aggregate stability, 30 months after application at 47 t ha<sup>-1</sup> of green waste biochar produced at 550 °C on a sandy loam soil.

Compared to other studies of soil physical parameters, there is limited comparable information on biochar impact on soil saturated hydraulic conductivity (Ks) (Castellini et al. 2015). Soil Ks governs water infiltration and solute movement within the soil profile, thus influencing the likelihood of soil surface runoff after a heavy rainfall or irrigation event (Omondi et al. 2016). Biochar produced using mesquite wood (*Prosopis sp.*) at 400°C (average rate of 133 t ha<sup>-1</sup>), decreased by 92 % and by 67 % the Ks of very permeable organic soil, but increased that of less permeable soils by 328 % (Barnes et al. 2014; Githinji 2014). Laird et al. (2010) observed no change in Ks of soil with intermediate permeability (repacked fine loamy soil), 500 days after incorporating biochar made from slow pyrolysis of hardwood (*Quercus* and *Carya* spp.) applied at rates of 0, 5, 10, and 20 g-biochar kg<sup>-1</sup> soil. Available water content (AWC) of soil is a key property in tropical climates because it contributes to reduce plant water stress. If biochar is able to increase soil water reserves in agricultural soils, it may be possible to reduce irrigation frequency and volume.

Biochar was reported to mainly improve the AWC of poorly structured soils. Glaser et al. (2002) noted an increase of 18 % in AWC on Terra Preta soils. Devereux et al. (2012) corroborated this result, reporting improved water retention through a change in soil porosity, pore size, bulk density and wetting ability, on repacked sandy loam soil amended with

biochar made from wood charcoal. Ouyang et al. (2013) obtained an increased in the AWC of 5.2 % for silty clay soil and 10.6 % for sandy loam soil, using a biochar made from dairy manure at a ratio of 2 % w/w in dry weight basis. However, Ventura et al. (2013) found no difference in soil water retention on a clay loam, two years after application of biochar made from fruit tree pruning residues using a traditional oven, at rates of 10, 30 and 60 t ha<sup>-1</sup>. Similarly, Ojeda et al. (2015) in a greenhouse experiment, indicated no influence of biochar on water retention of a sandy loam after 1 and 20 months, using six types of biochar produced from different biomass sources (pine, poplar or sludge) and pyrolysis processes (slow, fast or gasification), applied at a mean dose of 0.018 kg biochar kg<sup>-1</sup> soil.

## 1.2 *Biochar and soil chemical properties*

Soil pH is one of the fundamental soil properties influencing nutrient availability and many soil chemical processes (Hadi-Akbar Basri et al. 2013). Sanchez et al. (1983) observed that biochar increased the pH of amended soils by 0.4 - 1.2 pH units, with greater increases in sandy and loamy soils than in clayey soils. The short and long term implications of biochar on N immobilisation and mineralization are specific to soil-biochar interactions (Clough et al., 2013; Prommer et al., 2014). In some cases, biochar application could decrease soil N availability and plant tissue N concentration (Barbosa de Sousa et al. 2014; Bargmann et al. 2014). In other cases N and P uptake in corn plants grown in a sandy loam was increased after application of wood biochar but decreased in a silt loam soil (Yeboah et al. 2009). This is explained by the possible sorption of N by biochar (Reverchon et al. 2014) or immobilization of mineral N due to increased soil C/N ratio and input of labile C (Ippolito et al. 2014). Mitigation of N leaching loss following biochar addition reported by Zheng et al. (2013) was in part attributed to an increase in soil water holding capacity (WHC).

Reported mechanisms by which biochar can affect soil P content and plant uptake of P include: changing soil environment for microorganisms (Atkinson et al. 2010); alteration of soil P availability through anion exchange capacity (DeLuca et al. 2009); reduced P leaching due to sorption of both orthophosphate and organic P by biochar (Laird et al. 2010); and direct release of soluble P after application (Parvage et al. 2013). However, we noted inconsistent results as to whether biochar application enhances P sorption or its release. Enhanced P availability in biochar was reported to be greatly affected by pyrolysis

temperature regardless of feedstock; lower pyrolysis temperature biochar contained more potentially available P (Xu, Zhang, Shao, et al. 2016; Xu, Zhang, Sun, et al. 2016). Soil P availability is also influenced by interaction with the soil conditions and properties, e.g. retention time in soil, coexistence of other anions and nutrients on exchange sites and soil acidity. The incorporation of biochars to acidic soil at 40 g kg<sup>-1</sup> (4 %) reduced the sorption and increased available P. In calcareous soil, application of alkaline biochars (corn stover and switchgrass biochars) significantly increased the sorption of P and decreased its availability (Chintala et al., 2014). Phosphorus release by biochars was also found to be highly dependent on the presence of other cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Al<sup>3+</sup>, Fe<sup>2+</sup>) in the soil solution. Slow release was found to be due to the formation of precipitates between dissolved P and excessive Ca<sup>2+</sup> and Mg<sup>2+</sup> in an alkaline milieu (Qian et al. 2013), while Fe-P and Al-P bonds were observed in more acidic soils.(Xu et al. 2014). Biochar seems to be one of the most effective materials reducing soil K losses in regions with high rainfall (Widowati & Asnah 2014). Several studies reported soil exchangeable K increase after biochar application. This impact was in part due to a direct supply of K from biochar (Zong et al. 2016) or by indirect improvement in fertilizer use efficiency by adsorption of nutrients on exchange surfaces thus reducing leaching loss (Widowati & Asnah 2014).

From the cited literature, it is evident that the influence of biochar on soil physico-chemical properties is highly variable. Biochar effects on soil properties depend on factors including biochar properties (influenced by feedstock type, pyrolytic conditions), application rate, soil type, time after application and the interactions among these factors. Biochar appears to have more influence in coarse-textured soils, poorly drained or excessively drained soils, poorly structured soils and soils with low organic carbon content. Less influence is noted on soils containing high organic matter, in fine-textured and well-structured soils (Biederman & Stanley Harpole 2013; Burrell et al. 2016; Omondi et al. 2016). Few studies have evaluated the effect of tillage in interaction with biochar application, such as in the context of the common cultural system of furrows and ridges (FR) in Cameroon (versus flat ploughing, FP). Considering the former system is predominant in many underdeveloped countries (because of topography, small size of most farms in forested zones or the low mechanization level), and has been proven appropriate on humid soils (Ker, 1995), we investigated the effect of biochar in this context. We discuss how the addition of biochar affects physical and chemical

properties of an oxisol cultivated under two different tillage modes for corn production in Cameroon. Straw is also considered in our study since it is an agricultural residue generally buried in conjunction with the FR tillage mode, and which is proposed as a raw material for biochar production.

## **2. Materials and methods**

### **2.1 *Site description and irrigation system***

The study was conducted on an experimental field in the western highlands of Cameroon in Central Africa (5°36'52'' N, 10°16'85'' E) at 1418 m of altitude. The site is characterized by a typical weathered red soil with 5% slope, which had been under fallow for three years. The climate is tropical wet with a mean annual rainfall of 1850 mm mainly from March to October. Mean maximum and minimum temperatures are 29.4 °C and 12.9 °C. The soil has a clay loam texture (USDA 2014), with an acid pH of 5.8 and a relatively low bulk density. Detailed soil characteristics are presented in Tables 2 and 3. To ensure adequate soil moisture, an irrigation system was designed based on the following parameters: basic infiltration rate of the soil estimated at  $2.5 \times 10^{-4} \text{ m.s}^{-1}$  using the double ring infiltrometer method (ASTM-D5093, 2008), corn water requirements as per growing stages (FAO 2016), actual evapotranspiration, and soil water retention capacity. Water from a nearby river was pumped to irrigate the experimental plots by sprinklers, twice weekly during the dry season (first production period, from January to May 2014) and then occasionally, according to rain events during the rainy season (second production period, from July to November, 2014).

### **2.2 *Biochar production and characterisation***

Biochars used in this study were made from local organic residues, eucalyptus tree bark (EB) and corncob (CCB). They were manufactured using a locally-made retort kiln at a temperature of around 300 °C. Physical, chemical and biological parameters, of both CCB and EB were characterized (Djousse et al., 2017) using methods described in Table 4 and their characteristics presented in Tables 2 and 3.



**Table 2: Biochar and soil physical parameters**

Symbols	Parameters	Units	CCB *	EB*	Value at beginning ( $\pm$ CV)	Value at the end of first PP ( $\pm$ CV)		Value at the end of Second PP ( $\pm$ CV)	
						FR	FP	FR	FP
<b>Granular size parameters</b>									
/	0.05 < % < 2	%	97	89	40	42 $\pm$ 1	41 $\pm$ 1	42 $\pm$ 0	42 $\pm$ 0
/	0.025 < % < 0.05	%	3	10	26	24 $\pm$ 2	25 $\pm$ 2	26 $\pm$ 0	26 $\pm$ 0
/	% < 0.025	%	0	1	30	34 $\pm$ 1	34 $\pm$ 0	32 $\pm$ 0	32 $\pm$ 0
/	Texture	/		/				Clay loam	
MPD	Mean particle diameter	mm	0.24	0.13	/	/	/	/	/
UC	Uniformity coefficient	/	2.12	2.43	/	/	/	/	/
<b>Porosity related parameters</b>									
$\rho_a$	Bulk density	g cm <sup>-3</sup>	0.33	0.46	0.76 $\pm$ 10	0.75 $\pm$ 9	0.72 $\pm$ 13	0.80 $\pm$ 9	0.77 $\pm$ 9
$\rho_s$	Particle density	g cm <sup>-3</sup>	1.62	1.63	2.65	2.65	2.65	2.65	2.65
$\Theta$	Total porosity	m <sup>3</sup> m <sup>-3</sup>	0.79	0.72	71 $\pm$ 4	0.72 $\pm$ 3	0.73 $\pm$ 5	0.70 $\pm$ 4	0.71 $\pm$ 4
<b>Water related parameters</b>									
$\Theta_s$	Saturation water	m <sup>3</sup> m <sup>-3</sup>	/	/	0.68 $\pm$ 4	0.62 $\pm$ 1	0.74 $\pm$ 19	0.62 $\pm$ 16	0.72 $\pm$ 9
$\Theta_r$	Residual water	m <sup>3</sup> m <sup>-3</sup>	/	/	0.27 $\pm$ 11	0.21 $\pm$ 4	0.36 $\pm$ 32	0.18 $\pm$ 48	0.34 $\pm$ 24
AWC	Available water content.	m <sup>3</sup> m <sup>-3</sup>	/	/	0.04 $\pm$ 16	0.06 $\pm$ 3	0.08 $\pm$ 21	0.14 $\pm$ 29	0.15 $\pm$ 23
Ks	Saturated hydraulic conductivity	m s <sup>-1</sup>	/	/	2.1E-4 $\pm$ 9	/	/	2.4E-4 $\pm$ 58	4.1E-4 $\pm$ 70
/	Capillary rise	g g h <sup>-1</sup>	5.07	5.19	/	/	/	/	/
$\Theta_x$	Relative humidity sorption	g g h <sup>-1</sup>	6.17	6.14	/	/	/	/	/

**N.B:** CCB= Corncob biochar; EB= Eucalyptus biochar; PP= Production period; FP= flat plot; FR=furrow-ridges

\* Adapted from Djousse et al., (2017); / = Not available.

**Table 3: Biochar and soil chemical parameters before application, 6 and 9 months after application**

Symbols	Parameters	Units	Biochar		Soil (oxisol)				
			CCB*	EB*	Value at beginning	Value at the end of first PP		Value at the end of second PP	
						Treated	Control	Treated	Control
pH <sub>H2O</sub>	pH water	/	9.31	8.11	4.4 ± 0.03	5.4 ± 0.1	5.1 ± 0.0	5.4 ± 0.1	4.9 ± 0.0
EC	Electrical conductivity	S m <sup>-1</sup>	0.028	0.068	0.05 ± 0.1	0.04 ± 0.2	0.04 ± 0.2	0.11 ± 0.2	0.10 ± 0.2
(CEC)	Sum of cations	cmol(+) kg <sup>-1</sup>	28.55	24.24	12.7 ± 0.1	12.8 ± 0.3	12.5 ± 0.3	11.2 ± 0.3	10.8 ± 0.3
N	Total Nitrogen	g g <sup>-1</sup> x 100	0.88	0.47	0.07 ± 0.08	0.07 ± 0.1	0.06 ± 0.2	0.07 ± 0.2	0.06 ± 0.2
P	Exchangeable Phosphorus	cmol(+) kg <sup>-1</sup>	4.56	4.25	8.7 ± 0.1	7.79 ± 0.4	7.2 ± 0.5	4.89 ± 0.3	4.2 ± 0.4
K	Exchangeable Potassium	cmol(+) kg <sup>-1</sup>	26.47	7.54	0.07 ± 0.1	9.0 ± 0.8	5.7 ± 0.4	1.1 ± 0.4	0.9 ± 0.4
Ca	Exchangeable Calcium	cmol(+) kg <sup>-1</sup>	0.80	14.73	/	/	/	/	/
Mg	Exchangeable Magnesium	cmol(+) kg <sup>-1</sup>	0.78	1.01	/	/	/	/	/
Na	Exchangeable Sodium	cmol(+) kg <sup>-1</sup>	0.50	0.96	0.01 ± 0.0	1.5 ± 0.4	1.3 ± 0.2	0.8 ± 0.2	0.8 ± 0.0
OM	Organic matter	g g <sup>-1</sup> x 100	/	/	3.8 ± 0.1	8.8 ± 0.3	6.6 ± 0.3	10.3 ± 0.2	8.4 ± 0.4
/	Graphitic Carbon	g g <sup>-1</sup> x 100	37.7	24.9	/	/	/	/	/
OC	Organic carbon	g g <sup>-1</sup> x 100	29.7	27.8	2.2 ± 0.1	5.1 ± 0.3	3.8 ± 0.3	5.9 ± 0.2	4.9 ± 0.4
C/N	Carbon nitrogen ratio	/	76	112	30 ± 0.0	72.8 ± 0.3	63.3 ± 0.3	84.3 ± 0.3	81.7 ± 0.5

**NB:** Soil samples collected from the top 10 cm of soil; CCB= Corncob biochar; EB= Eucalyptus biochar; FR = Furrow-ridges plots, FP = Flat plots; PP= Production period.

\* Adapted from Djousse et al., (2017)

### 2.3 *Experimental setup*

The treatments were organized in a split-plot design, with the main plots being the soil tillage mode (FP vs FR system), and with the sub-plots being one of the four following treatments (T2 – T5) plus a control (T1). The sub-plots were 4 x 4 m, separated from each other by an alley of 0.8 m. Replicates were assured with three blocks set perpendicularly to the slope gradient. The control consisted of the incorporation of straw (T1) while the other treatments consisted of the addition of CCB (T2); EB (T3); CCB and straw (T4); and of EB and straw (T5). All the plots received local standard fertilization, which consists in manual application of NPK (20-10-10) at the rate of 200 kg ha<sup>-1</sup> with urea (46-0-0) at 100 kg ha<sup>-1</sup>; these rates are used by farmers in the locality. The land was tilled using a rotor cultivator for FP and a hoe for FR. Due to its hilly landscape and the small size of agricultural plots, farmers in the region principally use this latter method. Straw from grasses present in each plot was either buried (T4 and T5) or removed (T2 and T3). For FR, grasses were pulled out with a hoe, then either kept aside, or partially buried in the furrow by applying a layer of soil over top. Two weeks later, biochar and fertilizer were applied manually on the entire surface of ridges, then immediately covered at a depth between 10-15 cm with a second layer of soil, in order to prepare the seedbed. Each plot had three ridges of 1 m each, spaced 50 cm apart. For FP, we first ploughed using the rotor cultivator at 10-15 cm depth, then two weeks later, biochar and fertilizer were manually spread on the entire surface of the plot and a second plough immediately completed to bury biochar and fertilizer, and to prepare the seedbed for sowing.

Improved corn seeds (PANNAR 12<sup>TM</sup>) were sown manually at about 4 cm depth, at a density of 4 plants m<sup>-2</sup> (50 x 60 cm in ridges and 50 x 65 cm in FP). The plots were irrigated when necessary as described in section 2.1. After harvesting (five months later), the agricultural residues were removed from the field and plots were ploughed using the hoe for ridges and the rotor cultivator for FP surfaces. Ridges were not moved to form new ones, but were instead disturbed and remained in the same position. A second corn production period of five months was then completed on the same plots, without application of either fertilizer or biochar, as generally done by farmers in the locality.

#### 2.4 *Soil sampling and analysis*

Soil samples were collected three times during the experiment: before ploughing, at the end of the first corn production before the second ploughing (6 months after treatment application), and at the end of the second production period (6 months after the second ploughing). For chemical and textural analysis, soil samples were collected between 1 and 10 cm depth, while a 100 cm<sup>3</sup> core was sampled for other physical analyses. For initial soil characterization, 12 undisturbed soil cores (4 per block) and 3 composite soil samples (12 sub-samples per block) were collected. At the end of the first production period, 30 soil cores (1 per plot) and 30 composite samples (4 random sub-samples per plot) were also collected following diagonal transects. A similar soil sampling was carried out at the end of the second production period. During this period, 30 composite soil samples were also collected at 2-week intervals to assess gravimetric soil moisture content. These composite samples were immediately placed in plastic bags after collection to avoid evaporation. These samples were analysed as described in Table 4.

#### 2.5 *Statistical analysis*

The data were analysed using the GLIMIX procedure of SAS followed by the Tukey HSD test for multiple comparisons. Analysis was carried out in two phases. First, the treatments T2, T3, T4, and T5 were compared to the control (T1) for the response variables. Second, the treatments were compared to each other, in order to interpret the effects of biochar type, soil tillage mode, production period and presence or absence of straw.

**Table 4: Summary of methods used for analysing biochar and soil**

Symbol	Names	Units	Methods	Equipment	References
<b>Biochar Physical properties*</b>					
$\rho_a$	Tapped bulk density	$\text{g cm}^{-3}$	Tapped density after 3 falls from 0.15 m	Cylinders	ISO 5311:1992, (1992)
$\rho_s$	Particle density	$\text{g cm}^{-3}$	Gas displacement pycnometer	(AccuPyc 1330 Micromeritics Norcross,GA, USA)	ASTM B923-10, (2015)
$\Theta$	Total porosity	$\text{m}^{-3} \text{ m}^{-3}$	$\Theta = 1 - \left(\frac{\rho_a}{\rho_s}\right)$	/	Flint and Flint, (2002)
	Particle size distribution	%	Mechanical and ultrasonic sieving	(RX-29, Ro-Tap, W.S. Tyler, Mentor, Ohio, USA) and an Allen Bradley sonic sifter	ASTM B923-10, (2015)
MPD	Mean particle diameter	mm	$MPD = \frac{\sum_{i=1}^{i=n} (R_i \times N_i)}{100}$ $R_i = \left(\frac{F_i}{S}\right) * 100$	/	
UC	Uniformity coefficient	/	$UC = \left(\frac{D_{60}}{D_{10}}\right) * 100$	/	ASTM D2862 - 10, (2010)
$\theta_{qx}$	Regression parameter for water sorption by capillarity under different tensions for 72 hrs	$\text{g g}^{-1}\text{h}^{-1}$	Tensions: -0.05, -0.25, -0.50, -0.75, -1 and -1.40	Tension table, non-linear regression	Adapted from Allaire and Parent (2004)
$\theta_x$	Total water sorption under different tension for 72 hrs	$\text{g g}^{-1}$	Tensions: -0.05, -0.25, -0.50, -0.75, -1 and -1.40	Tension table, non-linear regression	Adapted from Allaire and Parent (2004)
$\theta_g$	Moisture	$\text{g g}^{-1}$	Mass lost during 14 hrs at 105 °C	Muffle furnace	ASTM D1762-84, (1990).

Symbol	Names	Units	Methods	Equipment	References
<b>Biochar chemical properties*</b>					
pH	pH (H <sub>2</sub> O)	/	Electrode	VWR Symphony SP80PC pH meter	Rajkovich et al., (2011)
EC	Electrical conductivity	S m <sup>-1</sup>	Electrode	Radiometer Copenhagen CDM3 Conductivity Meter,	
CEC K, Ca, Mg, Na	Sum of the Exchangeable bases	cmol(+).kg <sup>-1</sup> cmol(+).kg <sup>-1</sup>	Calculated Agitation and filtration	sum of K, Ca, Mg, Na Optical Emission Spectrophotometry with inductively coupled plasma (ICP- OES) Optima 4300DV, Perkin-Elmer instrument G10 GYROTORRY shaker	
C <sub>tot</sub>	Total carbon	g g <sup>-1</sup>	Dry combustion, elemental analysis	LECO elemental analyzer	Adapted from Meng et al., (2014); LECO, (2009); Brewer et Brown, (2012)
CNSH- total	Total C, N, S and H	g g <sup>-1</sup>	Dry combustion	CNS-LECO Truspect	Brewer et Brown, (2012).
C <sub>org</sub>	Organic carbon	g g <sup>-1</sup>	Pre-treatment with hydrochloric acid, volatilization at 1380 °C and calculation	LECO elemental analyzer difference between the C <sub>graph</sub> plus C <sub>org</sub> and C <sub>graph</sub>	ASTM, (2002); CEAEQ, (2015); ISO, (2006)

Symbol	Names	Units	Methods	Equipment	References
$C_{\text{graph}}$	Graphitic carbon	$\text{g g}^{-1}$	Pre-treatment with nitric acid and volatilization at 1380 °C	LECO elemental analyzer	ASTM, (2002); CEAEQ, (2015); ISO, (2006)
<b>Soil Physico-chemical analysis</b>					
<b>Soil physical analysis</b>					
/	Texture	/	Bouyoucos hydrometer		Gee and Bauder, (1986)
/	Moisture content	$\text{g g}^{-1}$	Gravimetric	Oven	ASTM-D4959, (2014)
$\rho_a$	Bulk density	$\text{g cm}^{-3}$	Cylinder method		ASTM-D7263, (2009).
$\Theta$	Total porosity	$\text{m}^{-3} \text{m}^{-3}$	Calculated from $\rho_a$ assuming a particle density $\rho_s$ of $2.65 \text{ g cm}^{-3}$		Rühlmann et al., (2006).
$K_s$	Saturated hydraulic conductivity	$\text{m s}^{-1}$	Constant head permeameter with the undisturbed samples		Sarki et al., (2014)
/	Soil water retention curves (desorption)	/	Pressure plate, soil and moisture ink, USA. Model LAB023		Durner and Flüher, (2005)
/	Van Genuchten soil water retention curve parameters	/	Soil Water Retention Curve software (SWRC, version 3.00 beta,).		Piracicaba, SP, (2001).
/	Available water content (AWC)	$\text{m}^3 \text{m}^{-3}$	Calculated as the difference in water content at -0.3 and -100 m of tension using measured values		Mohanty and Mousli, (2000)

Symbol	Names	Units	Methods	Equipment	References
<b>Soil chemical analysis</b>					
pH-H <sub>2</sub> O	pH water	/	1:2.5 soil-water ratio after 16 hrs	CG822 pH-meter with combine pH-electrode	(Pauwels et al. 1992)
EC	Electrical conductivity	S.m <sup>-1</sup>	1:5 soil-water ratio	Conductivity meter	
N	Total Nitrogen	g g <sup>-1</sup> x 100	Salicylic acid digestion - Kjeldhal procedure; steam distillation techniques	UDK 129 Kjeldahl Distillation Unit	(Pauwels et al. 1992)
P	Available Phosphorus	mg.kg <sup>-1</sup>	Bray II	UV-VIS Spectrophotometer at 660 nm.	
K	Exchangeable Potassium	cmol(+) kg <sup>-1</sup>	Ammonium acetate method	Flame photometer	
Na	Exchangeable Sodium	cmol(+) kg <sup>-1</sup>	Ammonium acetate method	Flame photometer	
CEC	Cation exchange capacity	cmol (+) kg <sup>-1</sup>	Ammonium acetate method	Extractor and Titrator	Ross and Kettering, (2011)
OC	Organic carbon	g g <sup>-1</sup> x 100	Walkley and Black	Titrator	(Pauwels et al. 1992)

\* Djousse et al. (2017) adapted from Allaire et al. (2015)



### 3. Results and Discussion

#### 3.1 *Biochar and soil physical properties*

##### 3.1.1 *Bulk density and total porosity*

There was no significant effect of biochar treatment ( $p = 0.27$ ) or biochar type ( $p = 0.78$ ) on  $\rho_a$ , 6 and 12 months after its application (Table 2 and 5). Similarly, Rogovska et al. (2015) did not find effects on  $\rho_a$  three years after application of biochar made at 450 °C from mixed hardwood biochar (*Quercus* spp, *Ulmus* spp. and *Carya* spp.) applied at the rate of 9.8 and 18.4 t ha<sup>-1</sup>. Our results are in apparent disagreement with the work reported by Karhu et al. (2011) on agricultural soil, by Ventura et al. (2013) on a clay loam soil and a meta-analysis done by Omondi et al. (2016) on biochar-amended soils. All reported positive effects of biochar on  $\rho_a$ , over a wide range of biochar application rates, explained by the low  $\rho_a$  of biochar resulting in lower soil  $\rho_a$  after application. The  $\rho_a$  of our biochar ranged from 0.33 to 0.46 g cm<sup>-3</sup> while that of our soil was 0.76 g cm<sup>-3</sup>. This was quite low compared to  $\rho_a$  of mineral soils (1- 2 g cm<sup>-3</sup>) but closer to  $\rho_a$  of organic soils (< 0.5 g cm<sup>-3</sup>) (Hossain et al. 2015). The effect of biochar was thus expected to be weaker considering the bulk density of our soil (Verheijen et al. 2010). The  $\Theta$  was not affected by treatment, biochar type, and soil tillage mode (Tables 2 and 5). Once more, these results are dissimilar to many previous studies (Bhattarai et al., 2015; Omondi et al., 2016), all of which reported increased soil porosity after the addition of biochar from different sources.

These findings could be explained in part by either the initial high porosity of biochar, leading to an increase in total soil micro-pores, or an alteration in soil pore size distribution. In the present case, there was no difference between the initial porosity of our biochars (79 % for CCB and 72 % for EB) and that of the soil (71 %) (Tables 2 and 5), which might explain our observations. Indeed, our results are supported by Omondi et al. (2016). His meta-analysis reported that soil porosity was not significantly affected by addition of biochar in highly porous soils and at low and medium application rates (3.5 % to 4 % which is equivalent to 23-36 t ha<sup>-1</sup> based on our biochar bulk density and assuming incorporation at 20 cm depth). These rates were almost twice those used in the present experiment, and suggest that the studied oxisol might need higher doses of biochar with higher porosity to effectively alter the

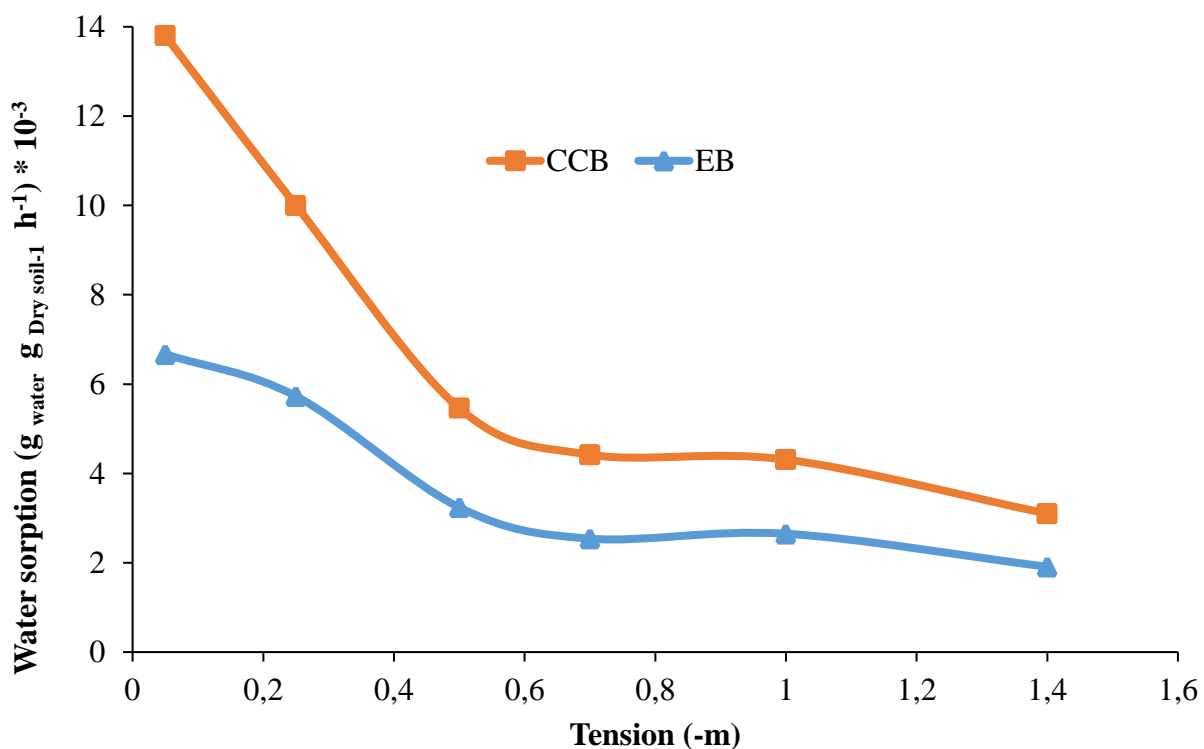
Θ. Production period and Soil tillage mode (STM) influenced  $\rho_a$  (Tables 2 and 5), values being higher during the second production period compared to the first; and in FP compared to FR. This is also in line with Θ that was lower during the second production period compared to the first ( $p=0.003$ ). The observed differences may be due to repeated tillage and to the effect of rainwater hammering, that together favour soil aggregate breakdown and compaction.

### 3.1.2 Saturated hydraulic conductivity (Ks), Available water content (AWC) and water retention curve parameters ( $\theta_s$ , $\theta_r$ )

We observed no change in Ks values during the experiment (Tables 2 and 5). Previous authors reported either a net short term reduction in Ks after application of biochar in sand and organic soils (Barnes et al. 2014; Githinji 2014), a net increase (Herath et al., 2013; Uzoma et al., 2011) or no effect (Castellini et al., 2015; Ouyang et al., 2013). A net increase was related to the high porosity of biochar, while a net reduction was attributed either to the initial hydrophobicity of biochar or to the creation of torturous interstitial space between sand and biochar grains. Our results could be explained by the low biochar application rate, since many experiments in which a change was observed were characterized by higher rates (Omondi et al. 2016). In addition, Herath et al. (2013) reported that generally poorly-drained soils exhibited a significant change in their Ks with biochar addition; this oxisol is well drained with high Ks ( $2.06 \times 10^{-4} \text{ m s}^{-1}$ ). The high variability of Ks values could also have contributed to hinder statistical differences between biochar-amended and non-amended plots. Biochar application had no significant effect on AWC ( $p=0.22$ ), independently of the type of biochar ( $p=0.76$ ), but production period did ( $p<0.0001$ ).

Hardie et al. (2013) also reported no significant effects of a green waste biochar applied at a rate of  $51.8 \text{ t ha}^{-1}$  on water retention curve parameters of a clay loam soil. Major et al. (2012) found no significant effect on either the water holding capacity or the Ks of a clay soil following wood biochar addition at the rate of  $20 \text{ t ha}^{-1}$ . At an application rate similar to the present study, Jeffery et al. (2015) indicated no improvement in soil hydrological function of a sandy soil after biochar application at  $10 \text{ t ha}^{-1}$ . Hence, the use of biochar at the equivalent rate of  $15 \text{ t ha}^{-1}$  may have also contributed to the observed lack of effect on hydrological function. In fact, some of the studies in which positive effects of biochar on soil hydraulic

properties were reported used biochar application rates that are not feasible for field scale operational applications, such as 50 t ha<sup>-1</sup> (Jeffery et al., 2015), 40, 80 t ha<sup>-1</sup> (Jones et al. 2010), 88 t ha<sup>-1</sup> (Gaskin et al. 2007) and 195 t ha<sup>-1</sup> (Yu et al. 2013). Similarly, many studies reporting positive effects of biochar were carried out in pot experiments or with repacked soils under controlled environments, which do not reflect the field situation of oxisols (Hardie et al. 2013). Our biochar was produced at a relatively low temperature (300 °C) and had a low water sorption by capillarity rise (1.9 % for EB and 3.1 % for CCB) at tension as low as -1.4 (Figure 4), compared to 86.3 % for Nuchar produced at high temperature (1500 °C) (Allaire et al. 2015). This char effectively absorbs water only when the soil is saturated (6.7 % for EB and 13.8 % for CCB) (Figure 4), much less compared to Nuchar (275 %). The CCB and EB thus had higher levels of hydrophobic compounds impeding uptake of water into pore space, especially during the first production period. Reduction over time of this hydrophobicity, in addition to the increase in soil organic matter content (Table 3), could explain the higher value of AWC obtained during the second production period compared to the first (p<0.0001).



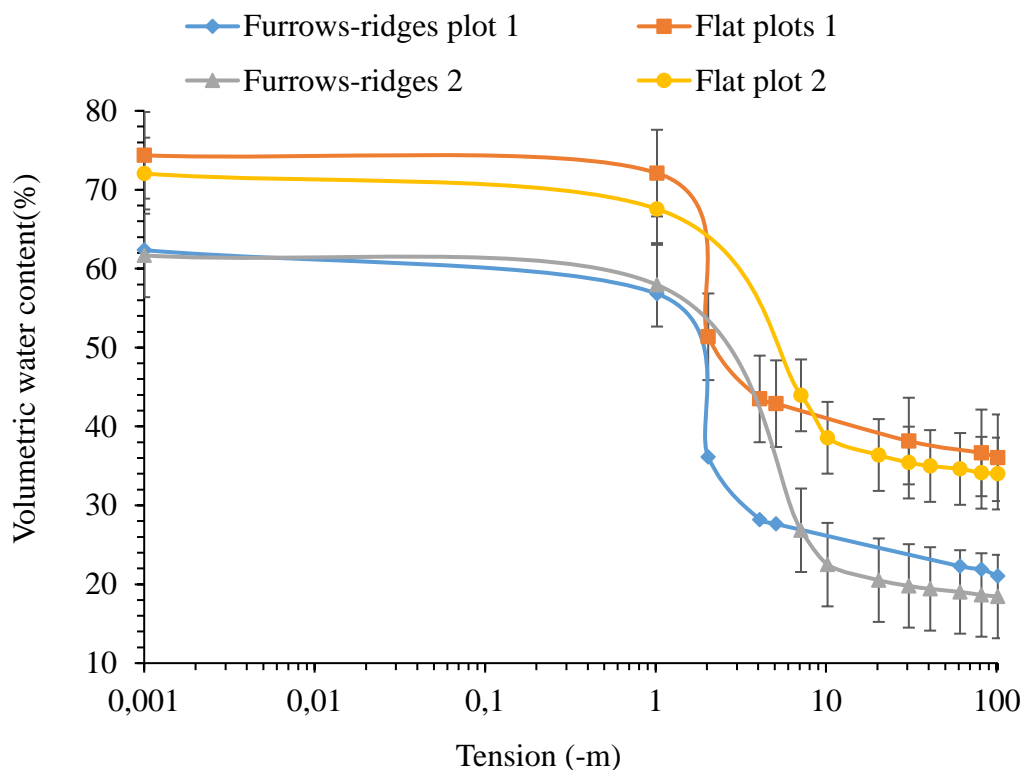
**Figure 4. Capillary rise of CCB and EB under tensions from -0.05 m (very wet) to -1.5 m (wet)**

**Table 5: Analysis of variance for soil physical parameters (degree of freedom and p-values)**

Parameters	DF	$\Theta$	$\theta_r$	$\theta_s$	$\rho_a$	AWC	Ks	$\theta_g$	
								DF	P
<b>Treatment versus control</b>									
Production period (PP)	1	<b>0.003</b>	0.22	0.15	<b>0.007</b>	<b>&lt;0.0001</b>	-	2	<b>&lt;0.001</b>
Treatment (T)	4	0.19	0.53	0.28	0.27	0.22	0.83	4	0.70
T * PP	4	0.81	0.60	0.74	0.85	0.79	-	8	0.47
Soil tillage mode (STM)	1	0.46	<b>0.005</b>	<b>0.0009</b>	<b>&lt;0.0001</b>	0.0072	0.19	1	0.045
STM * PP	1	0.96	0.96	0.55	0.72	<b>&lt;0.0001</b>	-	2	0.34
T * STM	4	0.99	0.65	0.19	0.65	0.005	0.33	4	0.60
T * STM* PP	4	<b>0.009</b>	0.05	0.56	0.60	0.13	-	8	0.52
<b>In between treatments</b>									
Biochar type (BT)	1	0.75	0.64	0.46	0.78	0.76	0.56	1	0.43
PP	1	<b>0.007</b>	0.39	0.09	<b>0.01</b>	<b>&lt;0.0001</b>	-	2	<b>&lt;0.0001</b>
BT * PP	1	0.82	0.77	0.89	0.84	0.37	-	2	0.46
STM	1	0.35	<b>0.0005</b>	<b>0.0006</b>	0.0002	0.08	0.34	1	0.17
BT * STM	1	0.69	0.87	0.62	0.7	0.90	0.63	1	0.17
STM * PP	1	0.71	0.99	0.80	0.87	0.0002	-	2	0.49
BT * STM * PP	1	0.18	0.15	0.34	0.26	0.44	-	2	0.72
Straw (S)	1	0.25	0.49	0.47	0.31	0.03	0.74	1	0.62
BT * S	1	0.45	0.23	0.11	0.50	0.78	0.85	1	0.73
S * PP	1	0.21	0.40	0.66	0.26	0.77	-	2	0.38
BT * S * PP	1	0.86	0.95	0.08	0.87	0.73	-	2	0.84
S * STM	1	0.82	0.51	0.25	0.81	0.0005	0.80	1	0.44
BT * S * STM	1	0.76	0.95	0.82	0.29	0.50	-	1	0.35
S * STM * PP	1	<b>0.0005</b>	<b>0.01</b>	<b>0.04</b>	0.2865	0.12	-	2	0.12

**NB:** DF = degree of freedom;  $\Theta$  = total porosity;  $\theta_r$  = residual water;  $\theta_s$  = water content at saturation;  $\rho_a$  = bulk density; AWC = available water content; Ks = saturated hydraulic conductivity;  $\theta_g$  = gravimetric water content. Significant p-values are in bold characters.

Soil water content (SWC) was not influenced by biochar type or its presence but varied from one sampling period to another (Table 2 and 5). This can be explained by the low water sorption capacity of our biochar as previously discussed (Figure 4) as well as the soil type (clay loam), which already has a good saturation water content related to its clay content. The greater SWC and AWC observed in FP compared to FR (Figure 5) can be explained by the fact that in FR, furrows act like drains, reducing the soil moisture in ridges. Fitted values of Van Genuchten parameters ( $\theta_s$ ,  $\theta_r$ ) are presented in Table 2, the  $\theta_s$  was only influenced by STM ( $p=0.0009$ ), values being higher in FP compared to FR, explanations are similar as those for  $\Theta$  and  $\rho_a$ . The  $\theta_r$  was not affected by the addition of biochar ( $p = 0.53$ ) nor by biochar type ( $p = 0.64$ ).



**Figure 5. Soil water retention curve as a function of treatment during the first and second production period (values  $\pm$  standard error)**

This was expected, as soil texture remains constant. Similar findings were reported by Uzoma (2011) with a biochar manufactured at 400 °C and applied at the rate of 10 t ha<sup>-1</sup> on a sandy soil and by Eastman (2011) and Laird et al. (2010) with an application rate of biochar up to 20 t ha<sup>-1</sup> on a loam soil. Tillage mode however positively affected  $\theta_r$  ( $p = 0.005$ ), values being higher in FP during both production periods (Figure 5). We hypothesize that organic matter

content builds up more quickly in FP (grasses were sliced up with the rotor cultivator and buried) compared to FR (grasses were buried). In summary, biochar influenced none of the measured soil physical properties, but tillage mode and production periods did affect these properties.

### 3.2 *Biochar and soil chemical characteristics*

Soil pH (Table 3) increased ( $p=0.001$ ), 6 and 12 months after biochar application independently of the soil tillage mode; the average value was 5.10 in the control and 5.45 in treated plots during the first production period, and increased from 4.95 to 5.38 during the second production period. Kannan Pandian et al. (2016) reported similar results with an increase in pH between 0.5 - 0.6 units after application of biochar made of *Prosopis* on an acidic red soil at the rate of 5 t ha<sup>-1</sup>. Several studies found that biochar addition may alter pH levels and the availability of soil nutrients such as Ca or Mg, while decreasing exchangeable Al<sup>3+</sup> and H<sup>+</sup> concentrations (Novak et al. 2009). Calcium and Mg were found to limit maize growth in highly weathered tropical soils (Major et al. 2010), or the availability of B and Mo, which are important cofactors in biological N fixation (Rondon et al. 2007). The EC of soil was not affected by biochar application, probably due to the dilution effect of soil and because the soil has yet, high levels of Al and Fe, causing higher initial EC ( $0.05 \pm 0.1 \text{ S m}^{-1}$ ) than the biochar ( $0.028 < \text{EC} < 0.068 \text{ S m}^{-1}$ ).

However, the soil EC value significantly increased during the second production period in all plots, imputable to the natural mineralisation processes occurring in the soil when tilled. FR exhibited higher values compared to FP possibly due to higher levels of mineralisable material (straw) compared to the FP tillage mode. No treatment affected CEC, despite the higher value of biochar CEC (24.24 cmol(+) kg<sup>-1</sup> for EB and 28.55 cmol(+) kg<sup>-1</sup> for CCB) compared to that of the soil (12.7 cmol(+) kg<sup>-1</sup>). This could be due to a dilution effect and leaching, since measurements were taken 6 months after biochar application. No or minimal changes in CEC were also observed after addition of pecan shell-based biochar at the rate of 40 t ha<sup>-1</sup> to a fine-loamy soil (Novak et al. 2009). Based on chemical analysis of biochar (Table 3), its application at the rate of 15 t ha<sup>-1</sup> was expected to contribute to additional N, available P and exchangeable K in the soil for at least one production period (Table 6). It was thus expected that soil N, P, K content of plots receiving CCB and soil P, K and Ca of plots

receiving EB will be different from that of control plots. This was not the case, 6 and 12 months after both types of biochar application (Table 7). This could be due to either rapid uptake by plants during the first production, to leaching or to sorption on biochar. Soil N and P content were significantly higher in FR plots compared to FP (Table 7), probably because added NPK fertilizer was buried in ridges, while it was mixed in the FP tillage mode.

**Table 6: Equivalent rate for biochar nutrient and carbon supply, maize needs and recommended fertilizer application rate**

Parameter	Units	CCB* (applied at 15 t ha <sup>-1</sup> )	EB* (applied at 15 t ha <sup>-1</sup> 1)	Recommended local mineral fertilization (200kg ha <sup>-1</sup> NPK + 100 kg ha <sup>-1</sup> N)	Maize needs for 6 t ha <sup>-1</sup> **	Maize needs for 3 t ha <sup>-1</sup> **
Nitrogen	kg ha <sup>-1</sup>	132	71	86	120	72
Phosphorus	kg ha <sup>-1</sup>	27	25	9	22	16
Potassium	kg ha <sup>-1</sup>	155	44	17	100	45
Calcium	kg ha <sup>-1</sup>	5	86	/	24	/
Magnesium	kg ha <sup>-1</sup>	5	6	/	25	/
Sodium	kg ha <sup>-1</sup>	3	6	/	15	5
Organic Carbon	kg ha <sup>-1</sup>	4455	4170	/	/	/

\* Adapted from Djousse et al. (2017b)

\*\*Adapted from FAO et al., (2003)

No change of soil available P after biochar addition was also observed in acidic soils in other studies (Chintala et al., 2014; Schneider and Haderlein, 2016; Zhang et al., 2016). A potential reason could be the fixation of P by Al, given the relatively low soil pH. Soil exchangeable K and Na were significantly lower at the end of the second production period compared to the end of first, probably due to nutrient uptake by maize plants or erosion. Similarly, Steiner et al. (2007) did not observe greater K availability after one cropping season when wood biochar was added to a Brazilian Amazon oxisol at the rate of 11 t ha<sup>-1</sup>. Biochar application increased soil organic carbon (Tables 3 and 7), explained by the high organic carbon content of biochar. The relatively high content of graphitic like carbon (Table 3) is also an indicator that applied biochar will remain stable for a longer period in these soils. In summary, the

biochar treatment positively affected soil pH and soil organic carbon, while both tillage mode and production period also affected several soil chemical variables.



**Table 7: Analysis of variance for soil chemical parameters (degree of freedom and p-values)**

Parameters	DF	N	P	K	Na	CEC	EC	pHwater	OC
<b>Treatments versus control</b>									
<b>Production period (PP)</b>	1	0.25	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	<b>&lt;0.0001</b>	0.12	<b>&lt;0.0001</b>	0.19	<b>0.0001</b>
<b>Treatment (T)</b>	4	0.11	0.51	0.64	0.36	0.95	0.12	<b>0.0001</b>	<b>0.01</b>
<b>T * PP</b>	4	0.49	0.30	0.46	0.36	0.96	0.46	0.85	0.31
<b>Soil tillage mode (STM)</b>	1	<b>0.03</b>	<b>0.02</b>	0.27	0.11	0.43	0.15	0.67	0.77
<b>STM * PP</b>	1	0.56	<b>0.01</b>	0.60	0.59	0.45	0.56	0.69	0.44
<b>T * STM</b>	4	0.27	<b>0.05</b>	0.67	0.74	0.98	0.06	0.37	0.72
<b>T * STM * PP</b>	4	0.43	0.13	0.64	0.85	1.00	0.75	0.24	<b>0.02</b>
<b>In between treatments</b>									
<b>Biochar type (BT)</b>	1	0.12	0.08	0.18	0.07	0.71	0.42	<b>0.0001</b>	0.68
<b>PP</b>	1	0.38	<b>&lt;0.00</b>	<b>&lt;0.00</b>	<b>&lt;0.00</b>	0.16	<b>&lt;0.0001</b>	0.42	<b>0.02</b>
<b>BT * PP</b>	1	0.12	<b>0.03</b>	0.14	0.12	0.59	0.73	0.45	0.66
<b>STM</b>	1	0.23	<b>0.0001</b>	0.47	0.26	0.29	<b>0.04</b>	0.48	0.56
<b>BT * STM</b>	1	0.95	0.08	0.74	0.88	0.75	0.10	0.36	0.37
<b>STM * PP</b>	1	0.26	<b>0.0001</b>	0.22	0.47	0.45	0.24	0.74	0.84
<b>BT * STM * PP</b>	1	0.14	0.06	0.80	0.67	0.62	0.79	0.77	0.22
<b>Straw (S)</b>	1	0.12	0.39	0.37	0.47	0.93	0.58	0.97	<b>0.03</b>
<b>BT * S</b>	1	0.74	0.89	0.74	0.88	0.82	0.36	0.60	0.96
<b>S * PP</b>	1	0.46	0.62	0.41	0.67	0.75	0.51	0.97	0.11
<b>BT * S * PP</b>	1	0.97	0.82	0.68	0.67	0.68	0.77	0.67	0.36
<b>S * STM</b>	1	0.10	0.06	0.74	0.32	0.81	<b>0.0001</b>	0.54	0.87
<b>BT * S * STM</b>	1	0.19	0.55	0.37	0.47	0.88	0.37	0.20	0.81
<b>S * STM * PP</b>	1	0.82	0.12	0.68	0.47	0.99	0.07	0.97	0.39

**NB:** DF = degree of freedom; **N** = total Nitrogen; **P** = exchangeable phosphorus; **K** = exchangeable potassium; **Na** = exchangeable sodium; **CEC** = cation exchange capacity; **EC** = electrical conductivity; **OC** = organic carbon. Significant p-values are in bold characters.

#### 4. Agronomic implications of the study

At the selected application rate of 15 t ha<sup>-1</sup> of biochar made from Eucalyptus bark and corncob residues (300 °C), biochar did not have an important influence on soil physical properties, but did have an effect on chemical properties, at the end of twelve months and two production periods of maize. The results have the following implications for farmers intending to use biochar for soil improvement.

- There is no drawback in using these biochars in oxisols under either tillage mode;
- The biochar did not affect water retention in these high porosity, low density and well-drained oxisols; biochar with a different particle size distribution might exhibit a different response;
- The tested biochars may be used to increase soil pH and organic carbon, with both studied tillage modes;
- The Furrow and ridges tillage mode contributed to better storage of soil total N and higher exchangeable K, compared to Flat ploughing;
- The use of straw instead of biochars in Furrow and ridges mode did not show any interest as far as soil water retention is concerned. Given the reported positive side effects of biochar, mainly its reported C sequestration potential (Wang et al. 2016), we recommend that straw be pyrolysed and the resulting biochar incorporated into soil instead of burying straw (as is actually done in Furrow and ridges tillage mode).

#### 5. Conclusion

Biochars made in a retort kiln at 300 °C, using local residues of eucalyptus tree bark and corncobs, and applied at the rate of 15 t ha<sup>-1</sup> on a clay loam soil in Cameroon (oxisol), markedly influenced only soil pH and organic carbon, and no other physical or chemical soil characters (after 12 months). Both biochars marginally increased  $\theta_r$ ,  $\theta_s$ , and AWC, but these effects were not statistically significant. The FP soil tillage mode was characterized by higher values of soil water content than FR. Total soil porosity was lower and water retention was higher in the second production period, compared to the first. The use of biochar of larger particle size and at higher application rates, as well as the assessment of the longer term fate of carbon from biochar, could constitute future research studies on these oxisols.

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### **Chapitre III**

#### **Biochar improves maize nutritional status and yield under two soil tillage modes, on an oxisol, Cameroon <sup>3</sup>**

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<sup>3</sup> Version intégrale d'un article publié : Djousse, K.B.M., Allaire, S.E., Munson, A.D., 2017. Biochar improves maize nutritional status and yield under two soil tillage modes. *International Journal of Science and Research*, 6(10):470-475

## **Abstract**

Our field trial in Cameroon assessed the effect of biochar on nutrient uptake and maize yield, following a single application of 15 t ha<sup>-1</sup> of biochar to an oxisol under two soil tillage modes. Treatments were (T2) fertilizer + corncob biochar (CCB); (T3) fertilizer + eucalyptus biochar (EB); (T4) Straw + fertilizer + CCB; (T5) Straw + fertilizer + EB and the control treatment (T1) consisted of fertilizer + straw only. Maize leaf tissues were collected at tasselling during each production period and analysed for macroelements. Biochar significantly increased maize yield by 55 % above control during the first production period and by 54 % during the second period. The CCB amendment positively interacted with the furrows and ridges (FR) tillage mode to improve yield during the first production period. By compositional nutritional diagnosis (CND), the most deficient nutrients were identified as P, Mg and K. Despite higher yield, maize plants in biochar-treated plots were more nutritionally unbalanced during the first production period compared to control plants; the inverse was observed during the second production period. Further studies are required to improve maize fertilisation recommendations, including biochar for continuous production on oxisols.

## **Keywords**

Oxisol, compositional nutrient diagnosis, biochar, plant nutrition, Cameroon, maize production.

## **Résumé**

Notre essai en champ au Cameroun a évalué l'effet du biochar sur le rendement et l'équilibre nutritionnel du maïs, suite à une seule application de 15 t ha<sup>-1</sup> de biochar à un oxisol sous deux modes de labour du sol. Les traitements étaient (T2) engrais + biochar de maïs (CCB); (T3) engrais + biochar d'eucalyptus (EB) ; (T4) Paille + engrais + CCB ; (T5) Paille + engrais + EB et le traitement de contrôle (T1) consistait en engrais + paille seulement. Les échantillons de feuilles de maïs ont été collectés immédiatement après la floraison pendant chacune des deux périodes de production pour la détermination en laboratoire de la teneur en macroéléments. L'application du biochar a significativement augmenté le rendement du maïs de 55 % par rapport aux parcelles témoin pendant la première période de production et de 54 % au cours de la deuxième période. Le CCB a interagit positivement avec le mode de labour en sillons-billons (FR) pour améliorer le rendement pendant la première période de production. Le diagnostic compositionnel nutritionnel (CND) a permis d'identifier le P, Mg et K comme nutriments les plus déficients. Malgré un rendement plus élevé, les plantes de maïs dans les parcelles traitées au biochar ont présenté un déséquilibre nutritionnel plus prononcé pendant la première période de production par rapport aux plantes témoins ; l'inverse a été observé pendant la deuxième période de production. D'autres études sont nécessaires pour améliorer les recommandations de fertilisation du maïs pour la production durable sur un oxisol amendé au biochar.

## **Mots clés**

Oxisol, analyse nutritionnelle compositionnelle, biochar, Nutrition foliaire, Cameroun, production de maïs.

## 1. Introduction

One of the environmental challenges for many tropical countries is to limit deforestation while increasing food production (Feller et al. 2001). Oxisols, predominant in the tropics, contain kaolinitic clay minerals and clay-sized oxides and hydroxides of metals such as iron and aluminium, few weatherable minerals, high levels of secondary minerals, low pH, deep profiles and high levels of available aluminium (Dabin 1984). Most of these properties contribute to reducing their capacity to retain plant nutrients. Over the past 10 years, there have been numerous studies of biochar application, which help to determine appropriate methods for biochar use to promote sustainable agriculture (Beesley et al. 2011; Cernansky 2015; Zhang et al. 2016). However, few studies have assessed the impact of biochar on maize production under different tillage modes. The interaction of biochar amendment with the furrow and ridges system, commonly applied by small farm holders to control erosion, potentially offers an opportunity to improve biochar use efficiency, since the biochar is concentrated spatially where most of the plant roots are located (in the ridge).

In order to better interpret the responses of maize to biochar amendment under different tillage modes, foliar analysis of the crop can be used as a diagnostic tool to detect nutrient deficiencies (Cottenie 1980; Magallanes-Quintanar et al. 2006; Parent et al. 2012). Compositional nutrient diagnostic (CND) is often used as an aid to assess the nutrient status of maize agro-ecosystems (Modesto et al. 2014). In fact, plant leaf nutrient composition is a unique consequence of plant adaptation to a particular nutrient environment under a given set of limiting factors (Parent 2011). Stress, resulting in growth disturbance, is related not only to deficiency of a particular nutrient, but also to inadequate relations among nutrients (Fageria et al. 2011). Prior experiments with biochar materials from bio-wastes have reported increased soil nutrient availability in highly weathered tropical soils and short-term increases in crop growth (Glaser et al. 2002; Steiner et al. 2008). For instance, Lehmann et al. (2003) reported an increased uptake of P, K, Ca, Zn, and Cu by cowpea (*Vigna unguiculata* (L.) Walp.) and rice (*Oryza sativa* L.) with higher charcoal additions on ferralsol and anthrosol. Major et al. (2010) successfully used leaf foliar analysis to identify Ca and Mg as deficient nutrients in a Columbian oxisol after biochar application.



The present study assesses the effect of two locally-produced biochars on maize nutrient uptake and yield under two common soil tillage modes. We hypothesized that the application of biochar at 15 t ha<sup>-1</sup> will: (i) result in a better nutritional status of plants in plots with biochar amendment; (ii) improve maize yield during the first two production periods.

## 2. Materials and methods

### 2.1 *Biochar properties and establishment of field experiment*

Two types of biochar were produced from local organic residues, eucalyptus tree bark (EB) and corncob (CCB), using the same pyrolysis process, a retort kiln at a temperature of 300°C, with average residence time of four hrs and gas recycling. These biochars were characterized in a previous study by Djousse *et al.* (2017a). Some of the main chemical properties of the chars, apart from those presented in Table 3, included alkaline pH (9.3 for CCB; 8.1 for EB) and relatively high ash content (10.1 % for EB; 5.3 % for CCB). The field trial was carried out from December 2014 to December 2015, in an agricultural farm in the western highlands of Cameroon in Central Africa (5°36'52'' N, 10°16'85'' E) at 1418 m altitude, with 5% slope. The plot was under fallow three years prior to the experiment. The soil is a clay loam with a pH of 4.9 and a low bulk density of 0.76 g cm<sup>-3</sup> (Djousse *et al.* 2017b). The climate is tropical wet with a mean annual rainfall of 1850 mm, principally over 8 months from March to October, and mean maximum and minimum temperatures of 29.4°C and 12.9 °C.

An irrigation system was designed and installed to ensure adequate soil moisture during the experimental period. The irrigation regime was based on basic infiltration rate of the soil estimated at 2.50x10<sup>-4</sup> m.s<sup>-1</sup> using the double ring infiltrometer method (ASTM 2008), corn water requirements as per growing stages (FAO 2016), actual evapotranspiration and estimated soil water retention capacity. Water was pumped from a river to irrigate the plots using sprinklers twice weekly during the dry season (first production period, from January to May 2014) and occasionally according to rain events during the rainy season (second production period, from July to November 2014). The experimental design was a split-plot with the main plots being the soil tillage mode (flat vs furrows and ridges system), and the sub-plots being one of the five treatments (T1-T5). Individual plot size measured 16 m<sup>2</sup>, spaced every 0.8 m. Three blocks set perpendicularly to the slope gradient were used as three replicates. The control treatment included fertilizer and the incorporation of straw, without

biochar (T1). Fertilization consisted in manual application of N-P-K (20-10-10) at the rate of 200 t ha<sup>-1</sup> and urea (46-0-0) at 100 t ha<sup>-1</sup>, which is a standard rate for local producers. The treated plots received fertilizer and CCB (T2); fertilizer and EB (T3); fertilizer, CCB and straw (T4); and fertilizer, EB and straw (T5). Both biochars were manually applied at a rate of 15 t ha<sup>-1</sup> (dry weight). This followed suggested application rates in the literature (Liu et al. 2013). The land was tilled using a rotor cultivator for flat plots (FP) and a hoe for furrow and ridges plots (FR). Biochars without straw (T2 and T3) and with straw (T4 and T5) were buried at 15 cm depth using a hoe in ridges and a rotor cultivator in FP. Each FR had 3 ridges of 1 m each spaced 50 cm apart.

Improved corn seeds (PANNAR 12™) were sown manually at about 4 cm depth at a density of 4 plant m<sup>-2</sup> (50 cm x 60 cm in ridges and 50 cm x 65 cm in FP). After harvesting (5 months later), the agricultural residues were removed from the field and plots were again ploughed using the hoe for ridges and the rotor cultivator for flat surfaces. Ridges were not moved to form new ones but rather disturbed and remade at the same location. A second corn production period of five months was then completed on the same plots, without application of either fertilizer or biochars. Weeding was done manually twice per production period and earthing up once during the second weeding. Plants were chemically treated once per production period as per standard method of farmers in the region, using Cypercal C720 EC™ (active component cypermethrine) to control caterpillars. This attack was not specific to a particular plot.

## 2.2 *Yield estimation, leaf sampling and analysis*

Maize ears were manually harvested from all plants in rows number 2, 4 and 6 for FP and 2, 4 and 5 for FR, to avoid edge effects. Ears were shelled by hand then weighed, dried in an oven at 60°C for 72 h, then weighed again. These data were used to estimate moisture content on a dry weight basis. Twenty-four maize plants were harvested from each plot, which is equivalent to a surface area of 6 m<sup>2</sup> given the density of 4 plants per m<sup>2</sup>. Grain yield was estimated as the ratio of dried maize grain over a surface area and conversion was done to obtain the final yield in t ha<sup>-1</sup>. Ten topmost fully-developed leaf samples were randomly clipped at their base at tasselling (Hochmuth et al. 2012) from ten healthy plants distributed throughout each plot, during the first and second production period. Leaves were then dried

at 60 °C, weighed, and finely ground. The obtained powder was acid digested (Parkinson and Allen, (1975), analysed for N content using CNS-LECO Trumac, then for P, K, Ca, and Mg contents using the ICP-OES Optima 4300DV (Perkin-Elmer instrument).

### 2.3 Statistical analyses

The effect of treatments on leaf chemical parameters were analysed using the GLIMIX procedure of SAS followed by the Tukey HSD test for multiple comparisons. Analyses were completed in two phases. First, the treatments T2, T3, T4, and T5 were compared to the control (T1) for the response variables (N, P, K, Ca, Mg and yield). Second, the treatments were compared to test the effect of biochar type, soil tillage mode, production period and presence or absence of straw. The compositional nutrient diagnosis of plant leaves (CND) was computed using the procedure outlined by Parent and Dafir (1992). The theoretical basis of the CND method is that plants with balanced foliar nutrition (i.e., CND-r<sup>2</sup> close to zero) will have a greater biomass and yield than plants with a foliar nutrient imbalance (Aitchison 1982; Parent et al. 1994). The full composition array of nutrient proportions in plant leaves forms a simplex (S<sub>d</sub>) made of d + 1 nutrient proportions including d measured nutrients and a filling value (R<sub>d</sub>) to sum up the dry matter concentration (%) to 100. It is defined as follows (d=5, in this case):

$$S_d = [(N, P, K, Ca, Mg, R_d) : N > 0; P > 0; K > 0; Ca > 0; Mg > 0; R_d > 0; \\ N + P + K + Ca + Mg + R_d = 100] \quad (6)$$

Where R<sub>d</sub> was computed as follows:

$$R_d = 100 - (N + P + K + Ca + Mg) \quad (7)$$

The nutrient proportions were considered scale invariant after dividing by the geometric mean (G) of the d+1 components (Aitchison 1982) as follows

$$G = [N \times P \times K \times Ca \times Mg \times R_d]^{\frac{1}{d+1}} \quad (8)$$

Row-centered log ratios (clr) for each nutrient was computed as follows:

$$V_N = \ln\left(\frac{N}{G}\right); V_P = \ln\left(\frac{P}{G}\right); V_K = \ln\left(\frac{K}{G}\right); V_{Ca} = \ln\left(\frac{Ca}{G}\right); V_{Mg} = \ln\left(\frac{Mg}{G}\right); \\ V_{R_d} = \ln\left(\frac{R_d}{G}\right) \text{ and } V_N + V_P + V_K + V_{Ca} + V_{Mg} + V_{R_d} = 0 \quad (9)$$

The observations were ranked in decreasing yield order, and a partition of the database between two sub-populations was iterated using the Cate-Nelson procedure (Khiari et al.

2001a; Magallanes-Quintanar et al. 2004; Salvatore 2013). The highest acceptable yield cut-off value across nutrient expressions was selected to ascertain that the minimum yield target for a high-yield subpopulation will be classified as high yield, regardless of the nutrition expression (Magallanes-Quintanar et al. 2006). The CND norms  $V_i^*$  and their corresponding standard deviation  $S_i^*$  were calculated as the means and standard deviation values of row-centered log ratios  $v_i$  of the high-yield specimens, where  $i = (N, P, K, Ca, Mg, R_d)$ . The CND indexes (nutrient disequilibrium indexes) noted  $I_i$  as for the  $i^{\text{th}}$  nutrient and the global nutrient disequilibrium index (CND- $r^2$ ) were computed as a standard variable using the CND norms  $V_i^*$  their corresponding standard deviation  $S_i^*$  and the mean values  $\bar{v}_i$  of row-centered log ratios  $v_i$  as follows:

$$I_i = \frac{\bar{v}_i - V_i^*}{s_i}; \quad (10)$$

$$\text{CND} - r^2 = \sum_{i=1}^6 I_i^2 \quad (11)$$

These indices were then used for diagnosis. The indices were first ranked in increasing order, from the most limiting to the most abundant. Negative CND indices represent nutrient limitation and the most negative value indicates the most limiting nutrient among a set of nutrients. Positive CND indices are an indication of nutrient excess and the largest value indicates the nutrient present in greatest excess. When the CND- $r^2$  is close to zero, nutrient requirements are balanced and maximum plant growth is expected (Aitchison 1982; Parent et al. 1994). All calculations were performed using the Microsoft Excel 2013 software.

### 3. Results and Discussion

#### 3.1 *Maize yield in response to biochar application*

Application of biochar increased maize yield by 55% above the control i.e. from 3.6 to 5.6 t ha<sup>-1</sup> during the first production period and by 54 % above the control i.e. from 1.3 to 2 t ha<sup>-1</sup> during the second production period (Table 8). During the first production period, the maximum yield obtained was 8.33 t ha<sup>-1</sup>, with T4 (CCB and straw) and T2 (CCB) treatments. This value is in agreement with the range of 6-9 t ha<sup>-1</sup> expected in the region with improved seeds. The minimum yield was 1.67 t ha<sup>-1</sup> on control plots with T1 (fertilizer + straw). During the second production period, the highest recorded yield was 3.75 t ha<sup>-1</sup> on plots receiving T4, while the minimum obtained was 0.42 t ha<sup>-1</sup> on control plots with T1 (Table 8). Cate

Nelson analysis (paragraph 3.2) also revealed that all high yields were recorded on biochar-amended FR (T4 and T2) during the first production period. Zwieten et al. (2010) reported increased maize yields of 2.2 t ha<sup>-1</sup> over the control with biochar-amended ferralsol. Crane-Droesch et al. (2013) and Major et al. (2010) also reported increases in maize yield due to biochar amendment ranging from 20% to 140% above the control on Colombian savannah oxisol. Recorded increases during our experiment, presented in Table 8 fall within this range.

The positive effect of biochar on yield was thus more important during the first production period compared to the second (p=0.03), potentially because of better availability of nutrients with fertilizer additions. Biochar also positively interacted with FR, contributing to a better yield during the first production period (p=0.08); the CCB interaction with FR was found to be the most effective (p=0.05). These results demonstrate that biochar addition benefits plant growth and improves maize yield on oxisol soil, during two consecutive production periods (Table 9). More specifically, CCB (T2 and T4) appears to be the most appropriate biochar amendment when used on FR plots.

### 3.2 *Nutrient concentration and Compositional nutrient diagnosis (CND) of maize leaves*

Following Cate Nelson analysis, during the first production period 87% of the population of maize was below the determined yield cut-off value of 6.77 t ha<sup>-1</sup>, the corresponding  $\chi^2$  value with 6 df was 2.52 compared to the calculated CND-r<sup>2</sup> value of 3.28. During the second production period, 93% of the maize population fell below the cut-off the yield of 3.67 t ha<sup>-1</sup> with a corresponding  $\chi^2$  value with 6 df of 1.85 while the calculated CND-r<sup>2</sup> value was 5.24. Therefore, the CND-r<sup>2</sup> distribution for maize did not follow  $\chi^2$  distribution as previously assumed (Khiari et al. 2001b; Magallanes-Quintanar et al. 2006). Similar results were obtained by Parent et al. (2009). During the first production period, average total N concentration of high (2.43 %) and low yielding (2.27%) plants, fell between the accepted range of 2.25 % - 3.50 % (Cornforth & Steele 1981), (Table 10). This presumes that nitrogen absorption by plants was not a problem, since initially supplied N (mineral fertilizer) was sufficient to meet crop needs during this first production period (Table 6).

**Table 8 : Minimum, maximum, average and standard deviation of yield of maize yield during two production periods**

Parameters	Production period 1					Production period 2				
	T1	T2	T3	T4	T5	T1	T2	T3	T4	T5
Treatments										
Yield (Maximum value in t ha <sup>-1</sup> )	5.00	8.33	8.00	8.33	7.00	2.50	3.67	2.58	3.75	3.42
Yield (Minimum value t ha <sup>-1</sup> )	1.67	4.17	2.50	3.33	3.33	0.42	0.75	0.58	0.42	1.08
Average yield (t ha <sup>-1</sup> )	3.61	6.11	5.22	5.83	5.08	1.33	2.14	1.76	1.77	2.38
Standard deviation (t ha <sup>-1</sup> )	1.11	1.46	1.77	1.75	1.41	0.80	1.19	0.78	1.28	0.78

T1= Fertilizer + straw; T2 = Fertilizer + CCB; T3 = Fertilizer + EB; T4 = Fertilizer + CCB + straw; T5 = Fertilizer + EB + straw

For the second production period, both high and low yielding leaf N concentration (2 %), fell below accepted range. In the field we observed that plants were less vigorous and yellowish during the second production period compared to the first. This is supported by CND indices (Table 11) which indicate leaf N deficiencies during the second production period. However, statistical analysis (Table 9) did not detect any difference in N concentration of maize leaf tissues irrespective of biochar treatment, biochar type, soil tillage mode or production period. Soil analysis also indicated no statistical difference in soil N concentration of treated and non-treated plots initially, and throughout the two production periods (Djousse et al. 2017b). This indicates no specific retention of the initially applied N due to biochar application or its immobilization due to the high soil carbon content. We thus argue for the contribution of other nutrients or parameters to explain the observed statistical difference in yield.

**Table 9: Analysis of variance (ANOVA) of maize yield and leaf nutrient concentrations, as influenced by the production period, the treatment and soil management (degree of freedom and p-values)**

N.B: Figures in bold are values significant at 10 % confidence level

Parameters	DF	N	P	K	Ca	Mg	Yield
Treatments versus control							
Production period (PP)	1	0.20	<b>0.10</b>	<b>&lt;0.0001</b>	0.56	0.68	<b>&lt;0.0001</b>
Treatment (T)	4	0.90	0.50	0.41	<b>0.01</b>	<b>0.01</b>	<b>0.01</b>
T * PP	4	1.00	1.00	0.47	0.50	0.84	<b>0.03</b>
Soil tillage mode (STM)	1	0.20	0.90	<b>0.10</b>	0.21	0.76	0.67
STM * PP	1	0.90	1.00	0.38	0.50	0.25	0.11
T * STM	4	0.80	0.60	0.55	0.62	0.42	<b>0.08</b>
In between treatments							
Biochar type (BT)	1	0.60	0.30	0.11	0.17	0.57	0.29
PP	1	0.30	<b>0.0003</b>	<b>&lt;0.0001</b>	0.54	0.70	<b>&lt;0.0001</b>
BT * PP	1	0.90	0.60	0.21	0.62	0.94	<b>0.05</b>
STM	1	0.30	0.70	0.13	0.28	0.86	0.65
BT * STM	1	0.30	0.40	0.12	0.81	0.26	0.91
STM * PP	1	1.00	0.40	<b>0.07</b>	0.72	0.20	<b>0.09</b>
Straw (S)	1	0.70	0.70	0.98	0.39	0.69	0.89
BT * S	1	0.40	<b>0.09</b>	<b>0.07</b>	0.18	0.85	0.40
S * PP	1	1.00	0.80	0.12	0.32	0.24	0.46
S * STM	1	0.40	0.40	<b>0.10</b>	0.24	0.49	0.17

**Table 10 : Optimum leaf nutrient concentration for maize in New Zealand and USA (other studies), compared to high yielding plant leaf nutrient concentrations of the present study**

Nutrient	Unit	New Zealand Maize*	United States Maize*	Production period 1		Production period 2	
				Treated plots	Control plots	Treated plots	Control plots
<b>N</b>	mg g <sup>-1</sup>	2.25 - 3.30	2.76 - 3.50	2.43 ± 0.10	2.44 ± 0.09	2.15 ± 0.25	2.13 ± 0.44
<b>P</b>	mg g <sup>-1</sup>	0.18 - 0.32	0.25 - 0.40	0.12 ± 0.02	0.12 ± 0.01	0.12 ± 0.02	0.12 ± 0.02
<b>K</b>	mg g <sup>-1</sup>	1.70 - 3.00	1.70 - 2.50	1.40 ± 0.11	1.36 ± 0.00	1.59 ± 0.11	1.43 ± 0.01
<b>Ca</b>	mg g <sup>-1</sup>	0.40 - 0.80	0.20 - 1.00	0.26 ± 0.01	0.16 ± 0.00	0.25 ± 0.00	0.15 ± 0.05
<b>Mg</b>	mg g <sup>-1</sup>	0.13 - 0.30	0.20 - 0.50	0.28 ± 0.02	0.18 ± 0.01	0.25 ± 0.01	0.19 ± 0.01

\* Adapted from Cornforth et Steele (1981)



The CND indices also indicated P deficiency in plants during the first production period, both in treated and control plots (Table 11). No difference in P leaf tissue concentration due to biochar addition or soil tillage mode was observed at 10% confidence level interval (Table 9). These are contrasting as initially supplied P (table 6) was adequate to meet plant needs; its absorption and use by the plant might have been hindered. Duncan (2002) and Ch'Ng et al. (2014) argued that in acid soils (pH 4.5 to 6) soluble inorganic phosphorus is fixed by aluminium and iron resulting in substantial lock-up of P.

Concentration of P in maize leaves significantly declined during the second production period, which may have contributed to the observed yield decrease. Leaf K concentration of maize significantly increased during the second production period compared to the first, independently of treatment ( $p < 0.0001$ ), but remained below the acceptable range of 1.70 % to 3 % (Table 10). The CND indices also showed that K was deficient during the first production period in all plots, but only deficient in control plots during the second production period (Table 11). As for P, initial K supply to soil was adequate (Table 6) and its deficiency could mainly be the result of poor absorption by plants. Tisdale et al. (1993) reported that important reductions in yield levels can occur when K concentration is inadequate to fulfil crop requirements. This is referred to as “hidden hunger”; as such, K deficiency could have contributed to the high percentage of low yielding populations in both production periods, and more specifically to the lower yield observed in control plots during the second period. Also during this production period, leaf Mg and Ca concentrations were significantly higher in maize leaf tissues of biochar treated plots compared to that of control plots ( $p = 0.01$ ). Similar results were reported by Major et al. (2010) who noticed a significant higher Ca and Mg contents of maize flag leaves at tasselling in treated plots using wood biochar. This is a result of biochar application (Table 6) since they were the only source of Mg and Ca to soil. These elements were thus well absorbed by the plants. However, Mg was highly deficient in treated plots, while Ca was deficient in all plots, with deficiencies being more pronounced in control plots (Table 11).

**Table 11 : CND indices values of treated and control plot for each production period**

Production period 1				Production period 2			
Treated Plots		Control plots		Treated Plots		Control plots	
Indices	Values	Indices	Values	Indices	Values	Indices	Values
<i><b>IP</b></i>	-1.10	<i><b>IK</b></i>	-0.89	<i><b>IMg</b></i>	-1.29	<i><b>IK</b></i>	-0.75
<i><b>IK</b></i>	-0.43	<i><b>IRd</b></i>	-0.46	<i><b>Ica</b></i>	-0.19	<i><b>IN</b></i>	-0.51
<i><b>IRd</b></i>	-0.20	<i><b>IP</b></i>	-0.25	<i><b>IN</b></i>	-0.07	<i><b>IRd</b></i>	-0.45
<i>Ica</i>	0.12	<i><b>IMg</b></i>	-0.01	<i>IK</i>	0.15	<i><b>Ica</b></i>	-0.32
<i>IMg</i>	0.85	<i>Ica</i>	0.13	<i>IRd</i>	0.28	<i>IMg</i>	0.00
<i>IN</i>	0.90	<i>IN</i>	1.87	<i>IP</i>	0.66	<i>IP</i>	0.88
<i><b>CND-r2</b></i>	<i><b>6.45</b></i>		<i><b>4.04</b></i>		<i><b>4.66</b></i>		<i><b>5.53</b></i>

$I_i$  = CND indexes (nutrient disequilibrium indexes) where i= (N, P, K, Ca, Mg, R<sub>d</sub>).

*N.B.* Deficient nutrients are in bold-italic; relatively to “0”, lowest values indicate most deficient nutrients and higher values indicate nutrients in excess

Data from Djousse et al. (2017b) show that soil pH and organic carbon are the only measured soil parameters that were statistically affected by biochar treatment. It is thus likely that these were the main soil parameters responsible for improvement in yield observed in biochar-treated plots, through increased availability of soil nutrients. Hadi-Akbar Basri et al. (2013) argued that availability of nutrients for plant uptake is directly related to soil pH; soil with acidic pH (3 - 5.5) has generally higher concentrations of heavy metals, Al and Fe toxicities and lower CEC. During the first production period, P and K were deficient in both treated and control plots but Mg was only deficient in control plots. During the second production period Ca and N were deficient in both treated and control plots while Mg was deficient only in treated plots and K deficient only in control plots. Therefore, Mg could have been the main critical nutrient that contributed to the yield increase observed between control and treated plots during the first production period. A K deficiency could have contributed to the lowest yield in control plots during the second production period.

#### 4. Conclusion

This study in an operational field trial demonstrated that a single biochar application at a rate of 15 t ha<sup>-1</sup>, to a nutrient-poor, acidic tropical soil (oxisol), improved crop yields during two cropping seasons by 54 % above the control treatment. This indicates that biochar could be a valuable asset for the management of agro-ecosystems in humid tropical regions, where subsistence agriculture is practiced frequently on highly-weathered oxisols. Mechanisms by

which biochar interacts with the oxisol may include improved pH and organic carbon, thus better availability of P and other cations such as Mg and K in the rooting zone. The CCB char in particular positively interacted with the furrow and ridges tillage mode after application in the first production period, contributing to a better yield compared to EB char; this effect was limited in time (not observed during the second period). Maize plants were globally nutritionally unbalanced in both treated and untreated plots; P, Mg and K were identified as the most limiting nutrients. This suggests a need for further studies aiming to improving current maize fertilization practices on oxisols. Also, a longer term study is needed to determine when the positive yield effects of biochar application decline, in order to program further amendments.

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## **Conclusions générales, limites et perspectives**

### **1. Conclusions générales**

Plusieurs publications récentes rapportent que l'utilisation du biochar peut influencer de façon positive, entre autres : la croissance et les rendements des plantes et leur état sanitaire. Cependant, bon nombre de ces travaux soulignent la nécessité de multiplier les recherches sur le biochar issu de différents types de biomasses ainsi que leur application dans de nombreuses régions du globe (Schmidt et al. 2000; Warnock et al. 2007). C'est dans cette vision que ce projet de thèse a été conduit, avec pour objectif principal d'améliorer les connaissances sur la production et l'utilisation du biochar en agriculture tropicale, comme moyen de lutte et d'adaptation aux changements climatiques. Il s'est focalisé sur les problématiques fondamentales en rapport avec les techniques de production de biochar et sa qualité, l'interaction du biochar produit avec les modes de travail des sols agricoles et les interactions sol-plante. Notre principale question de recherche était de savoir si le rendement de la culture de maïs sur sols ferrallitiques pourrait être amélioré sous diverses pratiques culturales locales en associant le biochar à la fumure minérale habituelle (NPK).

Dans le chapitre 1, il était question de produire et caractériser du biochar à partir des résidus locaux agricoles et forestiers (rafles de maïs, écorce de Eucalyptus). Nous avons conçu et construit un four de type « retort » avec du matériel local au prix de cent mille frc cfa (163 US\$). Il présente la particularité de pouvoir recycler au cours de son fonctionnement les gaz de synthèse pour maintenir le processus de pyrolyse, réduisant normalement les émissions polluantes. Deux types de biochars ont été produits avec les résidus agricoles (rafles de maïs, CCB) et forestiers (écorce de Eucalyptus, EB). Les biochars produits ont été caractérisés et les propriétés comparées avec les normes de l'EBC et de l'IBI. Les biochars ont rempli tous les critères de base définis par EBC et IBI pour la définition du biochar et sont acceptables comme biochar de classe 3 selon IBI ( $10 \leq \text{Corg} \leq 30$ ). Le four dans son état actuel peut déjà être utilisé pour stimuler une production plus propre de biochar à des fins agricoles dans les pays en développement, avec des effets positifs multiplicatifs potentiels pour les agriculteurs locaux. Une analyse plus approfondie des deux biochars a révélé des propriétés intéressantes pour la production agricole sur sols fortement altérés. Certaines d'entre elles incluent : un pH élevé et une bonne capacité tampon, une teneur élevée en P et Ca soluble, Mg et K et un bon

potentiel de rétention d'eau. De manière générale, les propriétés physiques des deux biochars étaient relativement similaires, mais les propriétés chimiques diffèrent. Le CCB présentait un contenu en carbone plus élevé que EB. En dehors d'une teneur élevée en K soluble, le EB avait des éléments solubles et une teneur en azote plus élevés. EB pourraient avoir plus d'effets à court terme pour la production agricole, tandis que CCB pourrait générer des effets à plus long terme pour la séquestration du carbone dans le sol, en raison de sa nature plus récalcitrante.

Dans le chapitre 2, il était question de quantifier l'influence de ces biochars sur les propriétés physiques et chimiques d'un oxisol sous deux modes de labour pour la culture du maïs. Les biochars à base de rafles de maïs et d'écorce d'eucalyptus appliqués à la dose de 15 t ha<sup>-1</sup> sur un oxisol au Cameroun, ont augmenté le pH et le carbone organique du sol après 12 mois. Ces derniers peuvent donc être utilisés pour améliorer le pH du sol et sa teneur en carbone organique indépendamment des deux modes de labour étudiés. En effet, la forte acidité des oxisols engendre les problèmes de toxicité aluminique conduisant à l'inhibition de l'absorption du phosphore, du calcium et du cuivre. Par ailleurs, ces sols ont des teneurs réduites en Corg et donc en matières organiques, ceci les rend plus vulnérables à l'érosion et réduit leur fertilité. Les parcelles labourées à plat (FP) ont présenté des valeurs plus élevées de teneur en eau du sol comparativement à celle labourées en sillon-billon (FR) ; ceci serait dû à l'effet drainant des FR. Le mode de travail du sol en sillon et billons a contribué à un meilleur stockage de N total des sols et à une teneur en K échangeable plus élevée par rapport au labour à plat. L'utilisation de la paille au lieu du biochar en mode sillon et billons n'a pas montré d'intérêt pour la rétention d'eau dans le sol, cependant l'augmentation du carbone organique du sol justifierait le choix d'utilisation du biochar en lieu et place de la paille comme c'est le cas actuellement. Sur le plan agronomique, il ressort donc qu'il n'y a aucun inconvénient à utiliser ces biochars dans les oxisols sous l'un ou l'autre mode de travail du sol et qu'au contraire, il améliorerait certaines propriétés du sol.

Dans le chapitre 3, on a évalué l'effet de deux biochars sur le rendement et l'équilibre nutritionnel des plants de maïs sous deux modes de labour du sol et durant deux saisons de production consécutives. Après une seule application de 15 t ha<sup>-1</sup>, les biochars ont effectivement augmenté le rendement du maïs en moyenne de 54 % par rapport au témoin

pendant chacune des deux campagnes de production. Cela indique que le biochar pourrait être un outil précieux pour la gestion des agroécosystèmes dans les régions tropicales humides, où l'agriculture de subsistance est pratiquée. Le type de biochar ainsi que le mode de travail du sol et la présence ou l'absence de paille n'ont pas influencé individuellement le rendement du maïs. Cependant il a été noté une interaction positive entre le biochar de rafles de maïs (CCB) et la campagne de production d'une part et d'autre part entre CCB et le mode de labour. Le CCB appliqué dans les billons a permis d'avoir de meilleurs rendements au cours de la première campagne de production. Malgré leur rendement plus élevé, l'indice de déséquilibre global (CND- $r^2$ ) révèle que les plantes dans les parcelles traitées étaient plus déséquilibrées pendant la première période de production par rapport au contrôle, l'inverse a été observé au cours de la deuxième période de production. Le P, le Mg et le K étaient les nutriments les plus limitants et correspondaient aux différences de rendement entre les parcelles traitées au biochar et non. Les mécanismes par lesquels le biochar a agi incluent l'augmentation du pH et du carbone organique du sol entraînant probablement une meilleure disponibilité du P et des cations tels que le Mg, Ca et K dans la zone racinaire (déficiences montrées par l'analyse foliaire). Nous n'avons pas pu valider ces augmentations dans le sol superficiel avec nos méthodes.

## 2. Limites

Les principales limites identifiées dans le cadre de notre étude sont les suivantes :

- ✓ Elle a été conduite sur un court terme et donc présente peu de connaissances sur la cinétique de dégradation du carbone organique dans le sol, pourtant déterminante pour la séquestration du carbone.
- ✓ Nous n'avons pas pu faire des mesures d'émissions des gaz du sol (planifiés, mais matériel défectueux) ; or la modification du pH et de la teneur en matières organiques affectent de nombreux processus biologiques dans le sol.
- ✓ Les effets du biochar sur les sols et la productivité des cultures restent spécifiques au type de biochar utilisé, à la culture et au type de sol amendé ; la généralisation des résultats obtenus devrait à cet effet être faite avec subtilité.
- ✓ Nous n'avons pas fait de bilan total du carbone ni d'études économiques encore moins d'acceptabilité sociale du biochar.

### 3. Perspectives

#### 3.1 *Perspectives locales*

Afin de mieux valoriser sur le plan local les résultats de notre étude, nos travaux futurs s'orienteront vers les points suivants :

- ✓ L'amélioration de la conception du four à pyrolyse afin de permettre une utilisation simultanée pour la pyrolyse et la cuisson des repas au niveau familial ;
- ✓ L'amélioration de son l'isolation thermique pour une meilleure efficacité énergétique, ce en vue d'atteindre des températures de pyrolyse plus élevées ;
- ✓ La diversification des tests sur la granulométrie, les taux d'application, et la fréquence d'application du biochar ;
- ✓ La quantification des émissions de gaz lors de la pyrolyse pour la réalisation d'un bilan total de carbone ;
- ✓ L'approfondissement des analyses foliaires afin d'aider à mieux préciser les formules de fertilisation plus adaptées pour nos sols (et en combinaison avec le biochar);
- ✓ Dans un contexte de valorisation à plus grande échelle, l'identification et le test d'autres résidus disponibles localement pour la fabrication du biochar.

#### 3.2 *Perspectives globales*

L'utilisation du biochar pourrait effectivement contribuer à réduire la déforestation et augmenter la sécurité alimentaire au Cameroun ou ailleurs ; en effet :

- ✓ Toute chose restant par ailleurs égale, pour un rendement de  $6 \text{ t ha}^{-1}$ , un producteur qui utilise le biochar aura besoin de 1.5 ha pour produire 12 t de maïs contre 2 ha pour celui qui ne l'utilise pas ; ce qui contribuerait à sauver environ 0.5 ha de forêts (réduction de la déforestation due à l'agriculture de 25 %).
- ✓ L'augmentation du rendement est en plus maintenue sur deux campagnes de production, donc les effets induits à moyen et long terme sur la réduction des quantités d'engrais chimiques (chers et nécessitant beaucoup d'énergies fossiles pour la fabrication) sont également escomptés.
- ✓ L'augmentation de pH (0,3 et 0,5 unités) et du carbone organique (0,4 %) rendent cette technologie acceptable pour le programme global « 4 pour 1000 » (initié par la France après la Cop 21).

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