

Article

Dry-Aggregate Stability and Soil Nutrients Responses to Reapplication of Biochar and Organic/Inorganic Fertilizers in Urban Vegetable Production

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Abstract: Background: Knowledge of the responses of soil aggregates to various fertilization practices can contribute to sustainable soil management in urban vegetable agriculture. Aims: The study assessed the soil fertility, dry aggregate stability, and aggregate total carbon (TC) and nitrogen (TN) retention after the reapplication of biochar, and organic/inorganic fertilizers. Methods: Four replicates of ecological sanitation (ECOSAN) manure, NPK fertilizer, corncob biochar (CCB), and CCB, combined with urea and cattle manure (UCF), were established in a randomized complete block design. Results: The application of ECOSAN increased the soil pH and also resulted in significant accumulation of available phosphorus (P), calcium (Ca), and magnesium (Mg). Compared to CCB treatment, UCF + CCB produced significant increases in the TC, TN, P, Ca, and Mg concentrations by 53, 140, 227, 27, and 78%, respectively, and additionally increased the 4.75–2.00 mm large macroaggregates and mean weight diameter. The total carbon increased significantly in microaggregates (>0.25 mm); macroaggregates (4.75–0.25 mm); and in the micro- and macroaggregate fractions of the ECOSAN, CCB, and UCF + CCB treatments, respectively. More so, the TN in micro- and macroaggregates was increased maximally by UCF + CCB and minimally by the CCB treatment. Conclusions: The reapplication of CCB had limited potential for carbon sequestration in soil aggregates, but its combination with UCF better reflects carbon and nitrogen stabilization in micro- and macro- aggregates, with greater potential in large macroaggregates.

Keywords: cattle manure; corncob biochar; NPK fertilizer; ECOSAN; nutrient retention

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1. Introduction

Urban agriculture prevails in a variety of agricultural systems around the world due to increased urban population and rural-to-urban migration [1]. Advantageously, urban agriculture favors the production of accessible and inexpensive fresh horticultural products, which, in turn, increases food security [2]. On the other hand, urban agriculture in developing countries has been associated with the production of ‘unhealthy vegetables’ containing high heavy metals concentrations from air pollution, compost piles, human excreta, and the use of untreated wastewater for irrigation [3,4]. Furthermore, unsustainable farming practice due to negligence to invest in a soil fertility improvement program as catalyzed by unsecured land tenure and the absence of legitimacy is among the problems in urban agriculture [3]. Nonetheless, urban agriculture improves the environment through the recycling of urban organics. Accordingly, plant nutrients are recovered and reused to maximize crop yields and farmers’ incomes. The sustainable management of organic/inorganic fertilization in continuous vegetable cropping systems is important in order to maintain the soil quality and ecosystem functions and services in urban agriculture.

Despite its potential environmental problems, inorganic fertilizers are used in some areas to improve urban crop yields. High levels of nutrient supplies and agricultural prod-

ucts characterize inorganic fertilizers; however, their lack of influence on soil organic matter (SOM) enhancement limits their effects on the soil structure. Reference [5] found a negative effect of NPK fertilization on the SOM content. On the contrary, organic fertilization appears to be an effective strategy in managing and maximizing the SOM content. The addition of organic fertilizers or in combination with inorganic fertilizers in vegetable production offers many possibilities to improve the resource use efficiency, nutrient retention, and preserve soil quality [6–8]. In addition to these and other studies on the urban horticultural system of Burkina Faso [9,10], the positive effects of various fertilization strategies on the soil quality and structure, especially on soil aggregate formation and stability, are still uncertain.

Organic matter (OM) plays a crucial role in the cycling of soil nutrients, biodiversity functioning, and soil structural characteristics. While SOM serves as an important cementing agent for the formation of stable soil aggregates, soil aggregates are indicators of the physical protection and turnover time for SOM [11]. Reference [12] found that the application of 70% pig manure to the soil increased the macroaggregates (2.00–0.25 mm) and aggregate stability indices (higher mean weight diameter (MWD)). The importance of soil aggregation in the context of SOM protection against decomposition remains important for proper management of the soil structure. The dry-stability of soil aggregates is a recognized decisive indicator of the soil structure influencing soil erosion by wind, root growth, etc. [13]. Despite numerous reports on the SOM parameters in relation to soil aggregation [14–16], there is a paucity of information on soil aggregation, as influenced by different fertilization strategies in horticultural systems. More so, little is known about the processes that govern carbon (C) turnover of the biochar amendment or other local fertilizers used by urban vegetable farmers in the Sudan Sahel of West Africa. Other studies [15–19] assessed the C retention capacity of water-stable aggregates (water-resistant) under different soil management systems, but the dynamics of the C and nitrogen (N) concentrations in dry-stable aggregates (wind resistant) that imitate the Sahel environment are limited.

There are numerous interesting reports in the literature on the positive and negative effects of added biochar on the soil quality [19–23]. An initial application of biochar to the soil usually leads to an increase in soil organic carbon (SOC), which seems to decrease over time [14,24,25]. A companion study showed a continued SOC decline, indicative of SOC loss over an initial corncob biochar (CCB) addition in a two-year rotational vegetable cultivation [7]. It has been found that the reapplication of biochar improves the soil structure more than its initial addition; however, both periods of application supported soil aggregate stability through increased soil pH and SOM content, and improved sorption capacity of the soil [14,26].

In recent times, considerable efforts have been made to explore ecological sanitation (ECOSAN) products as supplementary nutrient sources for soil fertility improvement. ECOSAN products have been used as soil amendments in West Africa, particularly Burkina Faso and Niger. The nutrients and OM in human excreta represent a valuable food resource for beneficial soil organisms that are involved in the recovery and recycling of nutrients for sustainable agriculture [27]. Reference [28] stated that the ECOSAN system can sustain livelihoods and reduce poverty in urban areas by returning nutrients from excreta to the soil to increase its fertility. Numerous studies have shown the effectiveness of ECOSAN products on some soil properties and crop yields [29–31]. Notably, [10] found that the total organic C and fertilizer value of the ECOSAN products were higher than that of inorganic fertilizer.

Assessing the contributions of various fertilization options to improving the C and N resilience in soil aggregates is crucial to promoting plant nutrition for sustainable vegetable production in a rapidly growing West African population. Such an assessment is important in a changing climate, as vegetable production in its reproductive stages is highly susceptible to increased disease prevalence and climate change [32]. Consequently, prevailing soil management practices must maintain soil productivity and withstand the effects of climate change. The study aims to determine the impact of the reapplication of

CCB alone and in combination with urea fertilizer and cattle manure (UCF) on a 2-year aged initial applications, including a 2-year application of NPK and ECOSAN fertilizers (i) on hierarchical dry-stable soil aggregates and mean weight diameter (MWD), (ii) on the concentrations and distribution of total C (TC) and total N (TN) in hierarchical dry-stable soil aggregates, and (iii) on the contribution of the different soil aggregate fractions to the TC and TN contents of a Haplic Lixisol. We hypothesized that (i) the soil nutrient retention, dry-stable macroaggregates, and aggregate TC and TN concentrations would be greater with co-application of CCB, organic and inorganic fertilizers compared to the sole addition of CCB, ECOSAN, or NPK fertilizers, and (ii) the large macroaggregate TC concentration will relate to the overall aggregate stability response due to the treatments.

2. Materials and Methods

2.1. Site Description

The experiment is an accompanying study that builds on a four-year (2014–2018) ‘Urban Food^{plus}’ project at Wayalguin in Ouagadougou, Burkina Faso. The study reported here was carried out in Ouagadougou (12°24′16.30″ N latitude and, 1°28′41.0″ W longitude), the capital of Burkina Faso. The area lies in the Sudano-Sahelian zone and is characterized by a semi-arid climate. A unimodal rainfall pattern with an average annual rainfall of 788 mm and a mean annual temperature of 28.2 °C prevail in the location. According to [7], the sandy loam textured Haplic Lixisol (Cutanic) soil is derived from floodplain sediments, and the plain topographic experimental site is situated on an elevated plateau in a floodplain that is outside of the flooding zones and groundwater fluctuations. The well-drained soil was previously used for intensive vegetable production; however, maize (*Zea mays* L.) was grown in order to homogenize the experimental area prior to the 2014 experimental setup. At the commencement of the experiment, the soil was moderately acidic (pH in CaCl₂ 5.9) and contained a SOC of 0.57%, TN of 0.06%, available phosphorus (P) of 135.8 mg kg⁻¹, potassium (K) of 29.6 mg kg⁻¹, calcium (Ca) of 940.1 mg kg⁻¹, magnesium (Mg) of 80.5 mg kg⁻¹, and cation exchange capacity (CEC) of 56.7 mmol_c kg⁻¹ with a bulk density of 1.60 g cm⁻³ [6].

2.2. Experimental Set-Up and Treatments

As reported by [6], the study was a multifactorial split-plot experiment that was comprised of a sequential vegetable cultivation under four soil fertility management practices, and in combination with two irrigation water qualities (untreated waste water or clean water), and two irrigation water quantities (farmers’ usual irrigation quantity (full) or two-thirds of the full irrigation (reduced)). The grown vegetables included lettuce (*Lactuca sativa* L.), cabbage (*Brassica oleracea* L.), amaranth (*Amaranthus cruentus* L.), jute mallow (*Corchorus olitorius* L.), roselle (*Hibiscus sabdariffa* L.), carrots (*Daucus carota* L.), pepper (*Capsicum* sp.), and okra (*Abelmoschus esculentus* L. Moench). During the first phase of the experiment (2014–2016), the soil fertility management practices consisted of an unfertilized control (CTL), the co-application of urea and cattle fertilizers (UCF), which constitute the farmers’ fertilization practice, corncob biochar (CCB), and the co-application of UCF + CCB. A total of 16 treatments combinations were replicated four times in blocks comprising plots of 2 m × 4 m (8 m²) and a distance of 0.7 m between plots.

The second phase of the study (2016–2018) was a split-split plot experiment in which half of the previous treatment plots were modified. Accordingly, CCB and UCF + CCB were repeatedly applied onto the same treatment plots, while the NPK and ECOSAN treatments were superimposed on 2-year-aged UCF and CTL plots, respectively. Based on the agronomic responses over the 4-year trial period (2014–2018), four fertilizer treatments (ECOSAN, NPK, CCB, and UCF + CCB) were selected and assessed, as described herein. These treatment plots received clean water irrigation during the first study phase, whereas waste water irrigation was carried out in accordance with the local farmers’ practice during the second phase of the study.

2.3. Treatment Description

The biochar used in the study was produced from corncobs, which is commonly available in the study location. The feedstock was air-dried and subjected to slow pyrolysis in a local kiln at 500 °C [8] and an Elsa barrel [33] for biochar production in 2014 and 2016, respectively. The corncob-derived biochar was manually crushed to <2 mm particles before it was manually incorporated into the soil at a depth of 0–20 cm with a hand hoe. The initial and repeated application of biochar was at the rate of 20 t ha⁻¹. The elemental contents of the biochar applied in 2014 were: pH (CaCl₂) 10.3, TC 683.6 g kg⁻¹, TN 8.8 g kg⁻¹, P 1.41 g kg⁻¹, K 3.30 g kg⁻¹, volatile matter 19.6%, and ash content 18.5% [7], while the pH (1:5 w/v deionized H₂O) 8.7 and TC 670 g kg⁻¹ characterized the biochar applied in 2016 [34]. Reference [8] noted that the N, P, and K inputs from the initial application of biochar amounted to 180 kg ha⁻¹, 28 kg ha⁻¹, and 66 kg ha⁻¹, respectively.

The ECOSAN fertilizer was a composed fecal amendment that was applied at a rate range of 16–23 t dry matter (DM) ha⁻¹. Prior to the application of ECOSAN, the analysis showed that the pH of the control soil was 6.48 and the concentrations of SOC, TN, P, K, Mg, and sodium (Na) were 0.64%, 0.06%, 131.44 mg kg⁻¹, 21.57 mg kg⁻¹, 67.35 mg kg⁻¹, and 30.63 mg kg⁻¹, respectively [34]. The application of urea fertilizer (CH₄N₂O, 46% N) was at a rate of 107 kg ha⁻¹, and the cattle manure was 12 t DM ha⁻¹ per crop. The application of NPK-SB (14-23-14-6-1) was at the rate of 1070 kg ha⁻¹ by surface broadcast. This was followed by working in the soil with a hand hoe and leveling of the soil surface with a leveling rake. The nutrient contents and quantities of the treatments are presented in Table 1.

Table 1. Elemental contents and quantities of the applied fertilizers.

| Treatment | Fertilizer Nutrient | Fertilizer Quantity ^a (kg ha ⁻¹) | Fertilizer Nutrient Concentration (g kg ⁻¹) |
|-----------------|---------------------|--|--|
| ECOSAN | C | 981.20–1373.00 | 6.10 |
| | N | 150.00 | 0.93 |
| | P | 114.20–159.80 | 0.71 |
| | K | 426.30–596.50 | 2.66 |
| | Ca | 416.60–583.00 | 2.60 |
| | Mg | 247.70–346.60 | 1.55 |
| NPK | N | 150.00 | 140.00 |
| | P | 47.30 | 58.08 |
| | K | 103.30 | 50.21 |
| Urea | N | 49.90 | 46.70 |
| Cattle manure | C | 1408.30–1439.50 | 11.73 |
| | N | 100.00 | 0.83 |
| | P | 53.30–54.40 | 0.45 |
| | K | 136.10–139.10 | 1.13 |
| | Ca | 67.50–69.00 | 0.57 |
| Corncob biochar | Mg | 15.40–15.70 | 0.13 |
| | C | 13,600.00 | 680.00 |

^a Reference [34]. ECOSAN, ecological sanitation fertilizer.

In accordance with the local farmers' discretion and crop needs, irrigation with watering cans was manually carried out once or twice per day. The applied waste water was sourced from the municipal canal, while clean water was from a local well or from a tap supplied by the National Institute of Water and Sanitation (Office National de l'Eau et de l'Assainissement, ONEA) Burkina Faso. The wastewater was rather dilute so that the nutrient and pollutant inputs were similar to those with clean water [8]. The quantities and the elemental concentrations of the clean and waste water inputs have been documented [6–8]. Other agronomic management practices, including weeding with hand hoes, were carried out in both phases of the study, following the local farmers' practice.

2.4. Soil Sampling and Analysis

After the 2018 crop harvest, undisturbed soil samples were collected from each individual treatment plot at a depth of 0–20 cm, corresponding to the depth of the soil tillage. The soil samples were air-dried at room temperature, packaged in covered plastic containers, and shipped to the Laboratory of the Department of Soil Science/Soil Ecology, Ruhr University Bochum, Germany, for analysis. A portion of the soil samples was sieved with a mesh size of 2 mm and used for the initial soil characterization; another part sieved with 4.75 mm mesh was fractionated into four different dry-stable aggregate classes.

2.4.1. Soil Chemical Analysis

The soil pH was determined in 1:2.5 soil to the CaCl₂ solution using a pH meter. Analysis of the TC and TN concentrations of the treated soils was by dry combustion (Vario EL Elementar Analysensysteme GmbH, Hanau, Germany), while the available P (Bray) was by photometric measurement (Perkin Elmer UV/Vis Spectrometer Lambda 2). The Na⁺, K⁺, Ca²⁺, and Mg²⁺ were extracted in NH₄Cl and measured by ICP-OES (Spectro Ametek-Spectroblue and Spectro Analytical Instruments GmbH, Kleve, Germany).

2.4.2. Aggregate Fractionation

Soil aggregates were separated by the dry sieving method [35] using a mechanical shaker (Retsch GmbH and Co. KG 5657 HAAN 1, West Germany). A 50 g air-dried, sieved soil (4.75 mm) was placed onto the topmost of three sequentially arranged sieves of 2.00, 1.00, and 0.25 mm and shaken at 45 revolutions per minute for 5 min. Subsequently, the four dry-stable aggregate fractions (large macroaggregates (4.75–2.00 mm = Lma), medium macroaggregates (2.00–1.00 mm = Mma), small macroaggregates (1.00–0.25 mm = Sma), and microaggregates, including silt and clay (<0.25 mm = Mia), were obtained, weighed, and expressed in a percentage (%) to the initial sample weight (Equation (1)). In addition, the MWD of the dry-stable aggregate fractions was calculated, (Equation (2)).

$$\text{DSA}(\%) = \frac{M_i}{\text{weight of soil (g)}} \times 100 \quad (1)$$

$$\text{MWD} = \sum_{i=1}^n X_i * W_i \quad (2)$$

where DSA = dry-stable aggregate of each size fraction, M_i = weight of each aggregate size fraction (g), MWD = mean weight diameter of aggregates (mm), X_i = mean diameter of each size fraction (mm), and W_i = proportion of the total sample weight occurring in the corresponding size fraction.

Following the measurement of the aggregate TC and TN concentrations, the contribution of each dry-stable aggregate to the TC and TN contents of the soil was computed for each treatment and the whole soil (Equation (3)).

$$\text{Soil aggregate contribution to TC content (\%)} = \text{DSA}_i * \text{TC_DSA}_i \quad (3)$$

where DSA_i = proportion of each dry-stable aggregate fraction (%), and TC_DSA_i = TC (or TN) concentration in the corresponding dry-stable aggregate fraction (%).

2.5. Statistical Analysis

The data collected from the various determinations were cleaned and subjected to one-way analysis of variance (in Randomized Blocks) using GenStat for Windows (Laws Agricultural Trust). The test for the significance of treatment means was generally at 5% probability, with the exception of the dry-stable aggregate and MWD data, which was tested at the 10% probability level. The mean separation was by least significant difference (LSD).

3. Results

3.1. Treatment Effect on Soil Chemical Properties

All of the chemical properties considered in the study were significantly ($p < 0.001$) influenced by the treatments (Table 2). The soil pH value increased to a maximum of 7.43 with the ECOSAN treatment, while the minimum pH value of 5.53 was obtained with the NPK fertilizer. The addition of CCB resulted in a slightly higher pH (6.67) than the co-application of UCF + CCB (6.34). The maximum TC concentration was found in ECOSAN and UCF + CCB-treated soils, while the minimum TC was observed with the NPK treatment. The ECOSAN and UCF + CCB treatments resulted in a higher TN accumulation than the individual application of NPK and CCB. More so, the soil C/N was higher with the addition of CCB > ECOSAN > UCF + CCB > NPK treatment. Soil available P was low with CCB but increased maximally with the ECOSAN > NPK > UCF + CCB treatments. Exchangeable bases (Ca^{2+} , Mg^{2+} , Na^{+} , and K^{+}) were significantly higher with ECOSAN than with the other treatments. The application of CCB and UCF + CCB also increased the Na^{+} concentration significantly more than NPK fertilization.

Table 2. Chemical properties of the amended soils following a four-year rotational vegetable cultivation.

| Treatment | pH in CaCl_2 | TC (%) | TN (%) | C/N | P (mg kg^{-1}) | Ca^{2+} | K^{+} ($\text{mmol}_c \text{ kg}^{-1}$) | Mg^{2+} | Na^{+} |
|---------------------|-----------------------|-------------------|-------------------|--------------------|---------------------------|---------------------|--|--------------------|-------------------|
| ECOSAN | 7.43 ^a | 1.17 ^a | 0.08 ^b | 14.10 ^b | 142.89 ^a | 259.98 ^a | 14.96 ^a | 52.15 ^a | 6.92 ^a |
| NPK | 5.53 ^d | 0.68 ^c | 0.08 ^b | 9.05 ^d | 139.37 ^a | 44.12 ^b | 1.10 ^b | 7.62 ^b | 1.97 ^c |
| CCB | 6.67 ^b | 0.88 ^b | 0.05 ^c | 16.78 ^a | 34.48 ^c | 46.13 ^b | 1.00 ^b | 7.26 ^b | 3.46 ^b |
| UCF + CCB | 6.34 ^c | 1.35 ^a | 0.12 ^a | 11.67 ^c | 112.72 ^b | 58.89 ^b | 0.73 ^b | 12.91 ^b | 3.26 ^b |
| SED | 0.11 | 0.09 | 0.01 | 1.04 | 7.13 | 9.38 | 1.55 | 3.58 | 0.43 |
| $\text{LSD}_{0.05}$ | 0.25 | 0.20 | 0.01 | 2.35 | 16.13 | 21.22 | 3.50 | 8.10 | 0.97 |

ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; UCF + CCB, urea and cattle fertilizer combined with corncob biochar; TC, total carbon; TN, total nitrogen; SED, standard error of differences of mean; and LSD, least significant difference at 5% probability level. Mean values with the same letter are not statistically significant at the 5% probability level, according to the analysis of variance.

3.2. Treatment Effect on Dry-Aggregate Stability and MWD

The proportions of dry-stable aggregates of the amended soils differed significantly in the 4.75–2.00 mm = Lma ($p \leq 0.10$) and 1.00–2.00 mm = Mma ($p \leq 0.05$) size fractions, while the proportions of the 0.25–1.00 mm = Sma and <0.25 mm = Mia size fractions were not significantly affected by the treatments (Figure 1). Accordingly, the mass of dry-stable Lma in UCF + CCB and NPK-treated soils was statistically similar, but the UCF + CCB amendment significantly increased the Lma more than the other treatments. While the ECOSAN, NPK, and CCB had statistically ($p \leq 0.05$) equal proportions of dry-stable Mma, ECOSAN and CCB significantly increased the proportion of Mma by 68.98% and 87.87%, respectively, compared to the UCF + CCB treatment. Nonetheless, the MWD of the UCF + CCB was significantly ($p \leq 0.10$) higher than the other treatments. In fact, the MWD increased by 21% and 19% with UCF + CCB compared to the ECOSAN and CCB treatments, respectively (Figure 2).

3.3. Treatment Effect on Concentrations of TC and TN in Aggregate Hierarchies

Among the treatments, the variability in the concentrations of TC and TN, and the C/N ratios in all the aggregate fractions, were mostly significant at $p \leq 0.001$ (Figures 3–5). The TC across the aggregate hierarchies and treatments ranged from 0.52 to 1.42% (Figure 3). Amongst the treatment, the aggregate TC concentration was almost the highest with UCF + CCB and lowest with the NPK input. While the TC concentration across the aggregate hierarchies was significantly increased with the UCF + CCB treatment, the TC was significantly higher in Lma than the other aggregate fractions with the CCB amendment. The highest aggregate TC concentrations were found in Lma with CCB and UCF + CCB, in Mma and Sma with UCF + CCB, and in Mia with the ECOSAN amendment. In comparison

to CCB, the TC concentration increased by 42%, 57%, and 52% in the Mma, Sma, and Mia fractions, respectively, with the application of UCF + CCB.

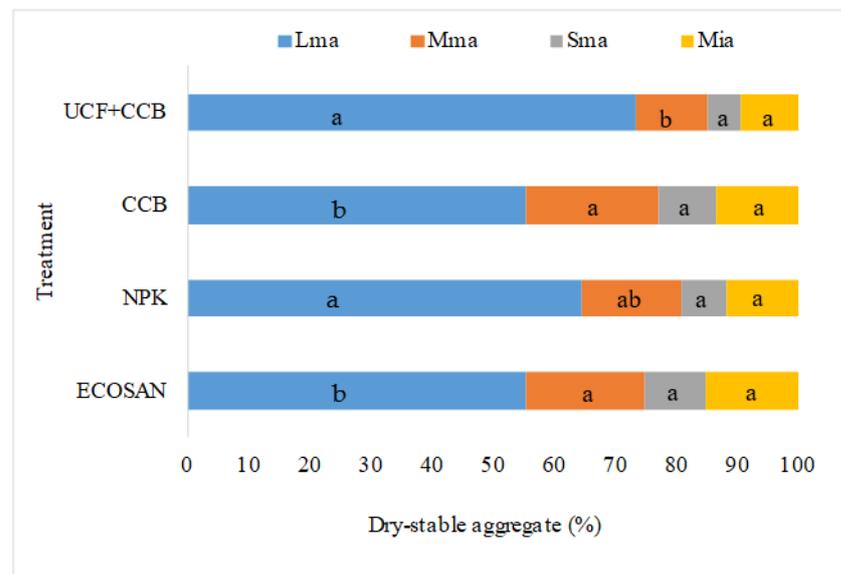


Figure 1. Dry-stable aggregate fractions of the amended soils. Lma, 4.75–2.00 mm large macroaggregates; Mma, 1.00–2.00 mm medium macroaggregates; Sma, 0.25–1.00 mm small macroaggregates; Mia, <0.25 mm microaggregates, including silt and clay; ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; and UCF + CCB, urea and cattle fertilizer combined with corncob biochar. The bars with the same letter are not statistically significant at the 10% probability level, according to the analysis of variance.

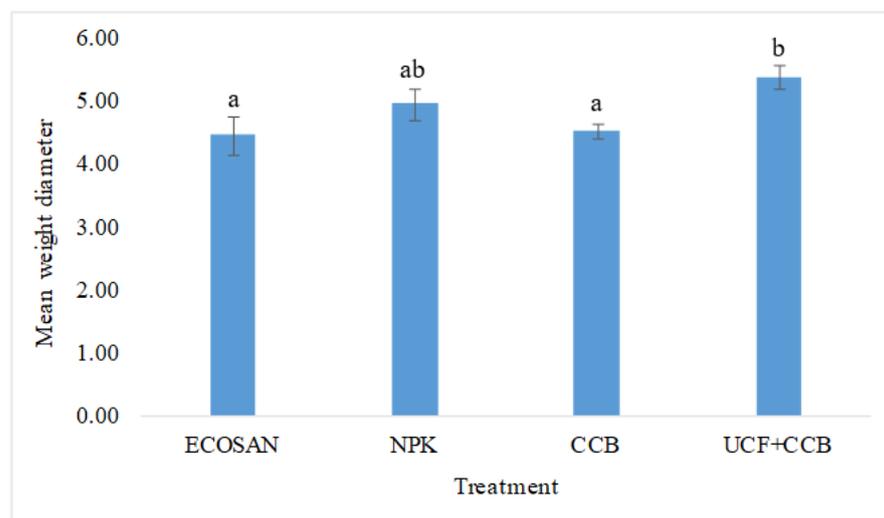


Figure 2. Mean weight diameter of the amended soils. ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; and UCF + CCB, urea and cattle fertilizer combined with corncob biochar. The standard error bars with the same letter are not statistically significant at the 10% probability level, according to the analysis of variance.

As presented in Figure 4, the aggregate TN concentration across the treatments and aggregate fractions ranged from 0.04 to 0.11%. Across the aggregate fractions, the application of UCF + CCB significantly produced the highest aggregate TN, while CCB gave the lowest. Although a similar trend of higher TN concentration in Mia > Sma > Lma > Mma was achieved with the ECOSAN and UCF + CCB treatments, almost all the treatments showed the highest and lowest TN concentrations in Mia and Mma, respectively. Remarkably, the

TN concentrations in the Lma, Mma, Sma, and Mia fractions were increased by 48%, 85%, 93%, and 73%, respectively, with UCF + CCB compared to CCB treatment.

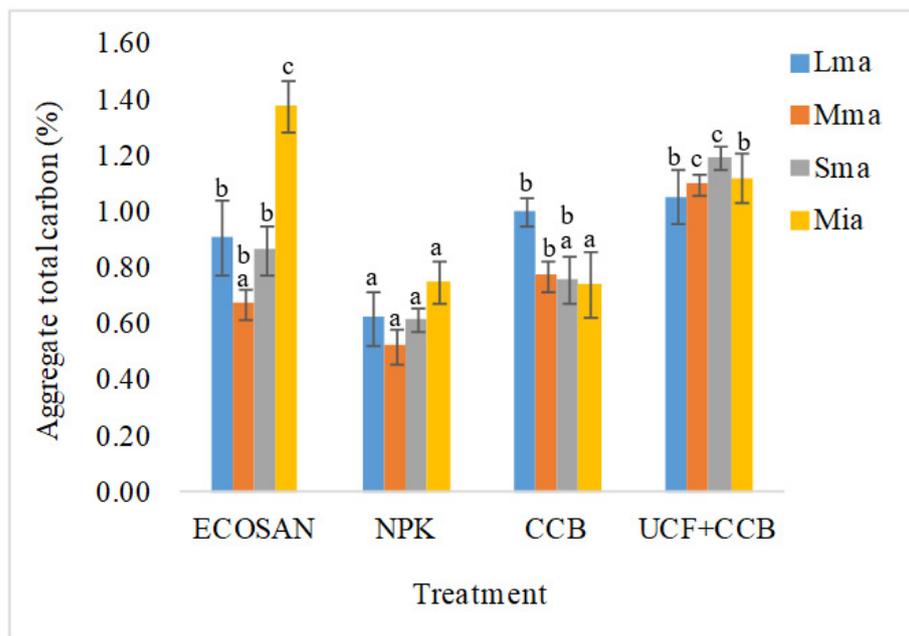


Figure 3. Total carbon concentrations in dry-stable aggregate fractions of the amended soils. Lma, 4.75–2.00 mm large macroaggregates; Mma, 1.00–2.00 mm medium macroaggregates; Sma, 0.25–1.00 mm small macroaggregates; Mia, <0.25 mm microaggregates including silt and clay; ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; and UCF + CCB, urea and cattle fertilizer combined with corncob biochar. The standard error bars with the same letter are not significantly different at the 5 % probability level, according to the analysis of variance.

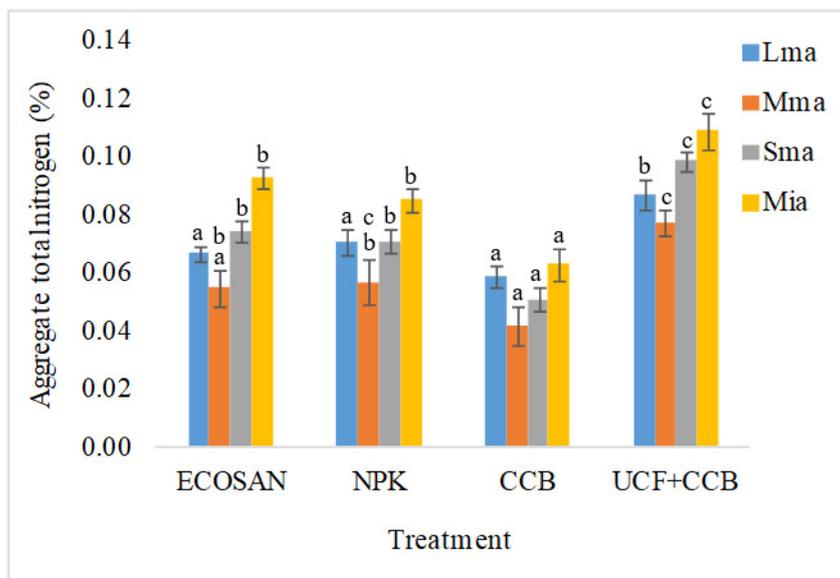


Figure 4. Total nitrogen concentrations in dry-stable aggregate fractions of the amended soils. Lma, 4.75–2.00 mm large macroaggregates; Mma, 1.00–2.00 mm medium macroaggregates; Sma, 0.25–1.00 mm small macroaggregates; Mia, <0.25 mm microaggregates including silt and clay; ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; and UCF + CCB, urea and cattle fertilizer combined with corncob biochar. The standard error bars with the same letter are not significantly different at the 5 % probability level, according to the analysis of variance.

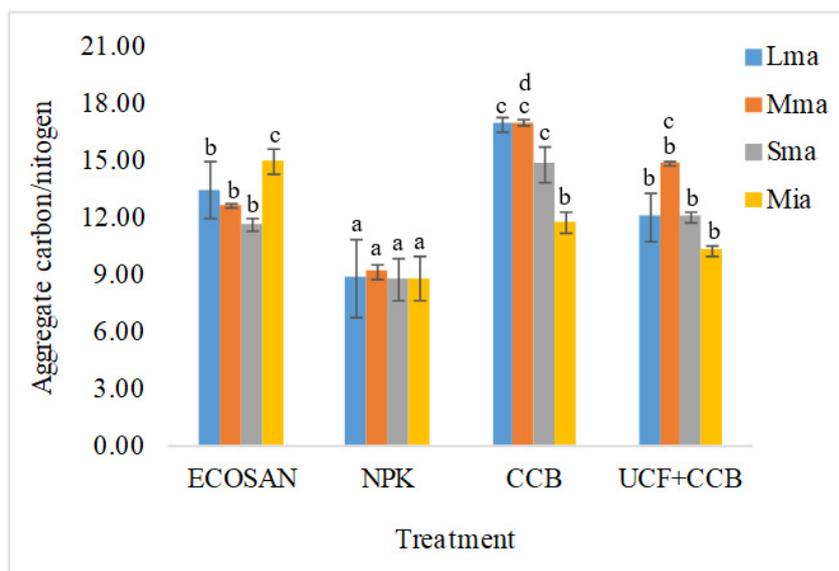


Figure 5. Soil carbon and nitrogen ratio of the dry-stable aggregate fractions of the amended soils. Lma, 4.75–2.00 mm large macroaggregates; Mma, 1.00–2.00 mm medium macroaggregates; Sma, 0.25–1.00 mm small macroaggregates; Mia, <0.25 mm microaggregates including silt and clay; ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; and UCF + CCB, urea and cattle fertilizer combined with corncob biochar. The standard error bars with the same letter are not significantly different at the 5 % probability level, according to the analysis of variance.

The C/N ratio across the aggregate fractions and the treatments ranged from 8.73 to 17.00 (Figure 5). While aggregate C/N with CCB was higher in the macro- than micro-aggregates, the opposite was the case with the ECOSAN treatment. As such, the C/N in the macroaggregates with CCB and in the microaggregates with ECOSAN was the highest amongst the treatments. The UCF + CCB treatment produced the highest and lowest C/N in the Mma and Mia fractions, respectively. Amongst the treatments, aggregate C/N was minimal and comparable with the NPK treatment.

3.4. Contribution of Soil Aggregates to TC and TN Contents of the Soil

The contribution of the soil aggregates to the TC and TN contents of the soils showed the highest and lowest contributions of Lma (50–72%) and Sma (6–11%), respectively, amongst the treatments (Table 3). Notably, the contribution of Lma to the TC and TN reserves was largely supported by the UCF + CCB (72%) and least of all by the ECOSAN (about 52%) amendment. Overall, Lma (63%) demonstrated a better association with the TC and TN contents than the other aggregate fractions combined.

Table 3. Percentage contribution of the soil aggregate fractions to the total carbon and nitrogen contents of the amended soils.

| Treatment | Lma | Mma | Sma | Mia | Lma | Mma | Sma | Mia |
|--------------|---|--------------|-------------|--------------|---|--------------|-------------|--------------|
| | Soil aggregate contribution to TC content (%) | | | | Soil aggregate contribution to TN content (%) | | | |
| ECOSAN | 53.97 | 14.11 | 9.44 | 22.47 | 53.32 | 15.46 | 10.89 | 20.33 |
| NPK | 64.62 | 13.78 | 7.28 | 14.31 | 64.83 | 13.33 | 7.40 | 14.44 |
| CCB | 62.01 | 18.73 | 8.15 | 11.11 | 59.26 | 16.47 | 8.91 | 15.36 |
| UCF + CCB | 72.12 | 11.81 | 6.26 | 9.81 | 72.17 | 10.06 | 6.26 | 11.51 |
| Grand mean * | 62.73 ± 17.09 ** | 14.56 ± 3.37 | 7.90 ± 1.71 | 14.97 ± 5.89 | 62.54 ± 1.39 | 13.29 ± 0.08 | 8.10 ± 0.12 | 14.97 ± 0.24 |

Lma, 4.75–2.00 mm large macroaggregates; Mma, 1.00–2.00 mm medium macroaggregates; Sma, 0.25–1.00 mm small macroaggregates; Mia, <0.25 mm microaggregates including silt and clay; ECOSAN, ecological sanitation fertilizer; NPK, NPK fertilizer; CCB, corncob biochar; UCF + CCB, urea and cattle fertilizer combined with corncob biochar; TC, total carbon; and TN, total nitrogen. * Grand mean of aggregate contribution to the TC or TN content. ** Standard deviation.

4. Discussion

4.1. Treatment Effect on Soil Chemical Properties

The reapplication of biochar and its integration with organic and inorganic fertilizers have previously been reported to increase the soil pH [14,22]. In contrast, however, a non-significant increase in pH (6.17 ± 0.23) due to the initial CCB application over a period of two years, as reported in a companion study [7], was similarly observed with the reapplication of CCB (pH 6.67) and UCF + CCB (pH 6.34) in the present study (Table 2). The lack of effect of these amendments on the soil pH could be assigned to its relatively low ash content. A high ash content and absorbent capacity of biochar are among the factors that determine its ability to increase the soil pH [36,37]. Nonetheless, the soil pH due to reapplied CCB and UCF + CCB is considered good for the growing of vegetables [38], since it favors nutrient uptake by plant roots.

On the other hand, an amendment with ECOSAN significantly ($p \leq 0.05$) increased the soil pH to 7.43 (slight alkaline), which is inconsistent with [29], who reported a non-liming effect (pH 5.86) after a two-year application of ECOSAN manure on a sandy loam soil. The alkaline effect of ECOSAN in this study relates to its highly alkaline content (Table 1), which replaced acid cations (Al^{3+} and H^+) at the exchange sites. Accordingly, an increased adsorption of largely improved K^+ , Na^+ , Ca^{2+} , and Mg^{2+} basic cations on the soil surfaces resulted in an increase in the pH of the ECOSAN-treated soil. The concentrations of Ca^{2+} and Mg^{2+} in ECOSAN-treated soils, which is considered excessively high for sandy soils [39], suggests impeded Ca and Mg uptake by the grown vegetables. Despite the fact that the observed pH with ECOSAN falls within the range (from 5.5 to 7.5) in which the growth of most vegetables is not inhibited [38], high levels of Ca in soils could be harmful to the growth and development of vegetables [39,40] and can also activate P adsorption on the surface of Ca carbonate (Ca-P) in alkaline soil [41,42], leading to P accumulation. In addition, the maximum increase in available P with ECOSAN ($142.89 \text{ mg kg}^{-1}$), attributable to the increase in the soil pH, allows for a low potential for P sorption by the Al and Fe oxides. This further suggests that much of the labile and absorbable forms of P were in excess of the needs of the cultivated vegetables. Consequently, the use of ECOSAN has been deemed unsuitable for continuous application in the study soil. Alternatively, ECOSAN could be used to reclaim acid soils, provided it is more economical than other lime materials that are expensive for most smallholder farmers. Compared to ECOSAN, the decline in the soil pH with NPK fertilizer is consistent with that of [7], who reported reduced soil pH due to continuous inorganic fertilization. The moderately acid pH of the NPK fertilized soil was mainly attributed to the nitrification of N. Another reason may be ascribed to the legacy effect from the UCF treatment of the previous 2 years. The influence of CO_2 from the decomposition of residual cattle manure-C may have contributed to the decline in the pH value. Decomposition of organic residues results in the production of organic acids that could potentially acidify the soil.

The repeated application of UCF + CCB resulted in better soil quality improvement compared to the CCB and NPK treatments. The maximum improvement in the TC, TN, and available P concentrations with UCF + CCB corroborates the findings of [26,43,44]; this observation is ascribed to the complementary contribution of each input that made up the treatment. While it is obvious that the biochar input was chiefly responsible for the quantifiable C benefit with UCF + CCB due to its high recalcitrant C contributing to a prolonged persistence in soils [45], the flux in the TN and P nutrients pools was mainly from the contribution of the UCF. The increase in the TN by 140% with UCF + CCB compared to CCB alone suggests the adsorption of NH_4^+ from urea and mineralized cattle manure-C onto the biochar surface, which probably could reduce the ammonification process and N leaching [46]. Besides the minimal biochar-induced N contribution, the biochar is capable of reducing the losses of N (NH_4^+ and NO_3^-), as its absorbent and large surface area can efficiently adsorb and retain ammonia in the soil [47]. It is presumed that the priming effect of the repeated addition of UCF + CCB often increases the decomposition rate of SOM due to high microbial activity and a higher release of availability energy. This possible

occurrence and the intensification of the CCB decomposition process by the addition of N (urea) could explain the increase in the TC concentration with the reapplication of the UCF + CCB treatment. The lower C/N obtained with UCF + CCB compared to the significantly higher C/N ($p \leq 0.05$) with CCB indicates a reduced microbial mineralization and, hence, reduced TN concentration. According to [48], an increase in C/N due to biochar application increases the microbial N limitation. The low TN concentration could also be linked to the low N content in CCB, which may be due to N volatilization during carbonization [49]. Nonetheless, the increase in TC with reapplied CCB, which agrees with the results of [26], was attributed to effective biochar-C storage due to its recalcitrant C. Regrettably, it is impossible to estimate the percentage increase in TC with CCB in the absence of a 'control' treatment, which also makes it difficult to assess whether the observed increase was mainly due to the quantification of initial or reapplied CCB amendment. Even though CCB has been reported to increase TC and TN in the tropical soil ecosystem [50], the remarkable increase in TC (+53%) and TN (+140%) due to UCF+CCB compared to CCB, distinguishes the former treatment for better fulfillment of C and N sequestration functions in the study soils.

Application of NPK fertilizer resulted in a higher accumulation of available P than the CCB and UCF + CCB treatments (Table 2). Reference [51] reported a similar result with NPK application over a two-year vegetable cultivation on sandy loam soil. The low available P concentration with reapplied CCB compared to UCF + CCB may be due to slow biochar mineralization and the associated P nutrient release. Co-application of the CCB with the farmyard manure compost, and NPK has been shown to significantly increase the soil available P compared to adding CCB alone [43,52]. Besides the contribution of biochar-P due to its ash content, the higher available P with UCF + CCB (+227%) than CCB came primarily from P additions via mineralized cattle manure and its retention, complemented by the ability of CCB to sorb nutrients. Additionally, the improvement in available P could also be attributed to the effect of increased base cations on reducing the P-sorbing constituents in the soil solution [53,54]. It thus implies that UCF + CCB can improve the level of inorganic P and also promote its retention against leaching losses.

The influence of the treatments on base cations was generally higher on the Ca^{2+} concentration compared to the other base cations (Table 2). A high content of Ca^{2+} in NPK fertilized soil was not unexpected, as residual cattle manure-C has a reputation for having unpredictable effects due to the high stability earlier alluded to. The high retention of Ca found in CCB-treated soil has been linked to a high exchange capacity and surface area, and direct Ca supply with biochar additions [55,56]. The high concentrations of Ca^{2+} and Mg^{2+} obtained with UCF + CCB were ascribed to a higher release of nutrients delivered into the soil by the treatment. Unlike in ECOSAN-treated soil, the levels of Ca and Mg in CCB and UCF + CCB amended soils were optimal for vegetables [39]. Likewise, the incomparable enhancement in TC and TN, and the optimally available P nutrient status is a clear indication that the reapplication of UCF + CCB could be an effective practice for improved and sustained soil fertility in a Haplic Lixisol. Given the high nutrient retention in the soil, it is reasonable to assume that the application of UCF + CCB on acidic soils would better meet the nutrient requirements of vegetables and thus promote continuous cultivation of the soil.

4.2. Treatment Effect on Dry-Aggregate Stability and MWD

The DSA distribution amongst the treatments showed a dominance of Lma ranging between 55 and 73% (Figure 1). The similarity in dry-stable Lma with NPK as UCF + CCB could be due to the residual effect of the previously added cattle manure, which, with repeated applications could have increasingly sustained the formation of the Lma compared to the change of land use system. The maximum improvement in the creation of dry-stable Lma (73%) by UCF + CCB was associated with; better aggregation through the supply of cementing agents via cattle manure [57]; positive involvement of TC, Ca and Mg nutrients

in increasing the formation of >2.00 mm soil fraction [58]; and biochar-induced aggregation due to its large surface area enhancing biochar-minerals complexes [59].

Garage [60] showed that the particle size of added biochar could determine the formation of macroaggregate in sandy loam soils. In view of the <2.00 mm CCB particles with a presumably large specific surface area, the improvement in Mma formation with CCB treatment could be related to a strong association of the CCB particles with the medium and small soil mineral particles. However, the considerable reduction in the proportion of Mma fraction obtained in UCF + CCB amended soil was likely due to the formation of Lma from Mma aggregates. Accordingly, the more pronounced effect of UCF + CCB in improving MWD by about 20% (Figure 2) compared to CCB and ECOSAN treatments indicates improved aggregate stability. It further implies improvement in soil structural stability, storage and stabilization of C, and movement of air, water and nutrients in the soil. Thus, the UCF + CCB amendment had the potential to reduce soil degradation through added organic matter, which could potentially help to reduce the impacts of wind erosion, a major threat to soils in the arid region. The insignificant ($p \leq 0.10$) increase in aggregate MWD after reapplication of CCB suggests that CCB alone could not effectively improve soil structural stability.

4.3. Treatment Effect on Concentrations of TC and TN in Aggregate Hierarchies

The reapplication of CCB induced a significant TC improvement ($p \leq 0.05$) in the macroaggregates but not in the microaggregates (Figure 3). This observation is in agreement with the report by [17]. This implies that the C sequestration potential of CCB was limited to macroaggregate fractions of the study soil. The significantly high C/N of macroaggregates (Figure 5), supporting a better protection of the CCB against mineralization, lends credence to this assertion. The low accessibility of biochar particles upon physical interaction with the organo–mineral complexes has been recognized [61]. Amongst the macroaggregate C/N data, the higher C/N of the Lma and Mma fractions than the Sma fraction reflects the maximum CCB protection due to the inhibition of CCB mineralization by these large and more stable aggregates. The maximum increase in TC concentration in Lma compared to the other fractions has been attributed to the binding of micro- into macro- aggregates by more labile SOM [62]. Apparently, organic C is protected from degradation via sorption on biochar large surfaces and within pores, and by the formation and stabilization of soil aggregates [17,21].

The concentration of TC in Lma was statistically similar with the UCF + CCB and CCB amendments. However, the percentage increase in TC by 42% (Mma), 57% (Sma), and 52% (Mia), respectively, with the application of UCF + CCB relative to CCB, resulted from the complementary contribution of the urea and cattle manure input. The improved TC concentration in all aggregate hierarchies with the UCF + CCB treatment was remarkable, suggesting a probable mechanism of induced C preservation by 'N fertilizer x cattle manure x CCB' amendment. Most studies have investigated the influence of biochar in combination with inorganic and/or organic fertilizers on the TC of bulk soils and not soil aggregates [19,43,57,63,64]. While the effectiveness of biochar in enhancing N-fertilizer use efficiency for plant growth [65] and the sorption capacity of biochar to affect plant residue-C and cattle manure-C may partially explain the observed response, the addition of N-fertilizer could facilitate biochar degradation processes in the soil [19,66]. As such, the overall aggregate TC delivered via UCF + CCB amendment indicates its superiority over the other treatments. The potential participation of all the aggregate fractions in promoting soil C sequestration implies a reduction in soil fertility depletion and mitigation of climate change.

Similar to aggregate TC, aggregate TN was significantly maximal in all aggregate fractions with UCF + CCB (Figure 4). It is noteworthy that the TN in the Lma, Mma, Sma, and Mia fractions improved by 48%, 85%, 93%, and 73%, respectively, with the reapplication of UCF + CCB compared to CCB. The aggregate C/N data indicated the complementary effect of UCF + CCB in moderating C mineralization and, hence, N release.

By providing a habitat to the bacteria involved in N retention [67], biochar can enhance the soil N retention. Reference [68] showed that adding CCB to sandy loam soil with repeated N treatment significantly reduced N leaching through better retention of organic N and NO_3^- -N. Our study result has established that, beyond the potential for increased C sequestration, UCF + CCB can also promote N sequestration in soils.

Across aggregate fractions, the effect of the treatments on TN was maximal, with $\text{UCF} + \text{CCB} > \text{ECOSAN} = \text{NPK} > \text{CCB}$ (Figure 4). The statistical similarity in the aggregate TN concentration with ECOSAN and NPK treatments was unexpected given the significant differences in their aggregate C/N data (Figure 5). For instance, the increased C/N in the Mia fraction with the ECOSAN amendment indicates that the organic C was less exposed to decomposition, which retarded denitrification process, leading to the N immobilization. The application of organic inputs in cultivated soils often contributes to C and N build-up in various aggregate hierarchies [69,70]. The aggregate TN concentration in NPK-fertilized soil was associated with increased SOM mineralization (evidenced by the minimal aggregate C/N) due to the effect of N fertilizer. The seemingly insignificant effect of the CCB on the aggregate TN concentration indicated that the CCB was more of a C- than N-source for the soil. Although the CCB was able to stabilize C in macroaggregates, the nitrification process was, however, impeded due to the stability and low accessibility of the entrapped biochar particles [61]. An initial application of biochar in a two-year field study significantly increased the TN in microaggregates [71].

4.4. Contribution of Soil Aggregates to TC and TN Contents of the Soil

A greater than 50% maximal association of Lma with the soil TC and TN contents was observed amongst the treatments (Table 3). The overall contribution of Mia to the TC and TN contents, which was about twice that of Sma, was attributed to the strongly adsorptive and reactive surfaces of silt and clay fractions and their deterministic control of microaggregates formation, serving as a habitat for C preservation [72]. In general, the soil is able to retain more TC (63%) and TN (63%) in Lma than in the other aggregate fractions combined. This indicates the importance of Lma in C and N sequestration in the soil. The most dramatic effect of UCF + CCB was its role in maximizing the proportion of dry-stable Lma and the concentrations of TC and TN in Lma, which favorably complemented its maximal association with the soil TC and TN contents by 72% each. Thus, while the TC in Lma follows the trend of $\text{UCF} + \text{CCB} = \text{CCB} > \text{ECOSAN} > \text{NPK}$, the association of Lma with the contents of the TC and TN reserves was of the order of $\text{UCF} + \text{CCB} > \text{NPK} > \text{CCB} > \text{ECOSAN}$ and $\text{UCF} + \text{CCB} > \text{ECOSAN} > \text{NPK} > \text{CCB}$, respectively. Considering the critical role of C in soil aggregate formation and stability, nutrient retention, and on climate protection, the huge contribution of Lma via UCF + CCB describes the treatment as a better option for improved soil sustainability.

5. Conclusions

The outcome of the study led to the conclusion that, after a 4-year consequential vegetable cropping, the corncob biochar reapplied to a Haplic Lixisol was a carbon rather than a nitrogen source, and its effect on improving the total carbon in large- and medium-sized macroaggregates and not in all aggregate fractions implies a limited potential for carbon sequestration in soil aggregates. Although the reapplication of the biochar in combination with urea fertilizer and cattle manure (UCF + CCB) would better promote carbon and nitrogen sequestration in all aggregate fractions, the 4.75–2.00 mm large macroaggregates have 72% greater sequestration potential than the other aggregate fractions combined. Additionally, the treatment improved the stability of the large macroaggregates by 33% compared to the biochar treatment alone, and could therefore be more beneficial against soil structural degradation, for example, by minimizing wind erosion, which is a major threat to semi-arid soils. In perspective, the ecological sanitation product as a soil amendment can offer the potential to reclaim acid soils through a significant improvement in base cations concentrations; however, the associated high accumulation of phosphorus,

calcium, and magnesium limits its suitability as a nutrient source for growing vegetables. In contrast, reapplication of the biochar + urea + cattle manure to soil can be considered an effective management practice for sustained soil chemical fertility (carbon, nitrogen, phosphorus, calcium, and magnesium) and soil structural stability. Therefore, the efficient use of UCF + CCB as agricultural inputs could provide the opportunity to support continuous urban agriculture.

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