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Impact of Different Agricultural Waste Biochars on Maize Biomass and Soil Water Content in a Brazilian Cerrado Arenosol

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Abstract: Arenosols in the Brazilian Cerrado are increasingly being used for agricultural production, particularly maize. These sandy soils are characterized by low soil organic matter, low available nutrients, and poor water-holding capacity. For this reason, adding biochar as a soil amendment could lead to improved water and nutrient retention. A greenhouse experiment was carried out using twelve biochars derived from four feedstocks (cotton husks, swine manure, eucalyptus sawmill residue, sugarcane filtercake) pyrolyzed at 400, 500 and 600 °C and applied at 5% *w/w*. The biochars' effect on maize biomass was examined, along with their contribution to soil physical properties including water retention, electrical conductivity (EC), and grain size distribution. After six weeks, maize plants in soils with eucalyptus and particularly filtercake biochar had higher biomass compared to those in soils with cotton and swine manure biochars. The latter's low biomass was likely related to excessive salinity. In general, our biochars showed potential for increasing θ in sandy soils compared to the soil alone. Filtercake and eucalyptus biochars may improve soil aeration and water infiltration, while applying cotton and swine manure biochars at levels <5% to avoid high salinity could contribute to improved soil water retention in Cerrado Arenosols.

Keywords: biochar; sandy soils; soil water retention; available water content; osmotic potential; particle size; porosity; Cerrado

1. Introduction

The Cerrado is the second largest biome in Brazil, covering 24% of the country's area [1]. The region's natural vegetation consists of a variety of vegetation types which vary structurally and in species composition [2]. The Cerrado is considered a global hotspot of biodiversity [3], in particular for plant diversity [4]. Yet, despite its ecological importance, the region has been experiencing rapid deforestation and conversion of natural grassland ecosystems since the 1970s, with replacement by exotic grasses (mainly *Brachiaria* sp.) for cattle-raising and conversion to arable croplands [2]. These land-use changes have significant environmental implications since they lead to lower carbon stocks, higher greenhouse gas emissions, lower evapotranspiration, and increased heat flux. The reduced

evapotranspiration can ultimately lead to decreased regional rainfall [2]. This regional climate change would greatly affect the local economy, including that of the state of Mato Grosso which encompasses a significant portion of the Cerrado. Maize has become an important crop for the economy of Mato Grosso, close behind soybean production, with total annual maize production in Mato Grosso exceeding 20 million tonnes in recent years [5]. The maize crop planted after the soybean harvest, in particular, accounts for 98% of all the maize grown in the state [5]. This crop is typically cultivated at the end of the rainy season (February), using residual soil moisture, and harvested in the dry season (usually in June). For this reason, dry season maize is particularly vulnerable to changes in precipitation patterns [6]. In addition, a large part of the maize is produced on Arenosols (sandy soils). Accounting for 13% of the area of Mato Grosso (about 11.7 million ha) [7], Arenosols are low in organic matter and their high sand content causes poor water retention [8], yet they are increasingly being used as cultivated soils. As their use for agriculture is of growing importance to the economy of Mato Grosso, sustainable management practices are necessary to improve the Arenosol's physico-chemical properties in order to increase their resilience to regional climate change.

Biochar (charcoal derived from waste biomass by pyrolysis) has been observed to increase soil water and nutrient retention under some conditions [9–12], particularly improving crop yields in tropical nutrient-poor soils [13], and so amending Arenosols with biochar could potentially be beneficial for this system. Biochar applications can affect soil hydrology by promoting mineral adsorption and increased soil aggregation, changes that may alter water flow in the soil [14]. Although several production factors influence the physical properties of the final biochar (e.g., heating rate, pressure, reaction vessel, pre-treatment, post-treatment, and other parameters), the temperature of pyrolysis is considered to be the most important determinant of a biochar's physical changes and thus its stability [15]. Microporosity of biochar has been demonstrated to increase with increasing temperature, contributing to greater water retention, though raising the temperature too high can cause loss of surface area and porosity [15,16]. Biochar's high porosity can lead to improved soil porosity, soil water content, plant available water content (AWC), soil bulk density, and soil hydraulic conductivity [17–20]. In addition, it can affect electrical conductivity (EC), with biochar EC tending to increase with increasing temperature of pyrolysis [21].

However, as with other agronomic contributions, biochar may also have either no effect or a negative effect on soil water and nutrient retention [19,22,23]. For this reason, it is necessary to more closely examine the soil physical properties related to soil hydrology that can be affected by biochar addition, including soil bulk density, porosity, and grain size distribution [23]. Understanding biochar's contribution to these soil physical properties is necessary in order to select appropriate biochars for the specific needs of a producer, in terms of their soil type and climate. The objective of our study was thus to better understand biochar effects of water retention on crop production in a Brazilian Cerrado sandy soil facing increasing agricultural use and regional climate change. A greenhouse experiment was conducted assessing maize growth in an Arenosol mixed with biochars produced from different local agricultural waste feedstocks heated at different temperatures of pyrolysis. In addition, physical properties of individual biochars and biochar-soil mixtures were examined to observe how they may alter soil water retention, hydraulic conductivity, and EC, in turn affecting maize growth. We hypothesized that higher temperature biochars would lead to higher water retention and nutrient content (as monitored by EC), and hence higher maize biomass.

2. Results

2.1. Maize Biomass

After 6 weeks, filtercake biochar had the highest mean above and belowground dry biomass at 600 °C, with dry aboveground biomass (16.7 ± 0.9) significantly ($p < 0.05$) higher than the control (11.8 ± 0.4) (Figure 1a). For both filtercake and eucalyptus biochars, mean aboveground biomass increased with increasing temperature, while for cotton and swine manure biochars mean plant biomass decreased with increasing temperature. Maize biomass was significantly ($p < 0.05$) lower in soils with cotton and swine manure biochars compared to eucalyptus and sugarcane filtercake biochars and the control (soil without biochar) (Figure 1). Aboveground dry biomass in soils with swine manure biochar at 400 °C was significantly higher than at 600 °C (Figure 1a). Aboveground dry biomass in eucalyptus biochar treatments did not differ between the temperatures (Figure 1a), and belowground dry biomass also showed no significant differences between the temperatures for any of the feedstocks (Figure 1b). Analysis of soil macronutrients showed that soils with cotton and swine manure biochars had the highest potassium (K) and sulfur (S) levels compared to the other biochars, but the lowest calcium (Ca) levels (Figure 2). Both K and Ca were strongly ($p < 0.001$) correlated with aboveground dry biomass: K negatively correlated ($R = -86$), while Ca positively correlated ($R = 0.74$) (Figure S1). The Ca:Mg ratio was also highest in eucalyptus and filtercake biochar treatments compared to cotton and swine manure biochars (Figure S2).

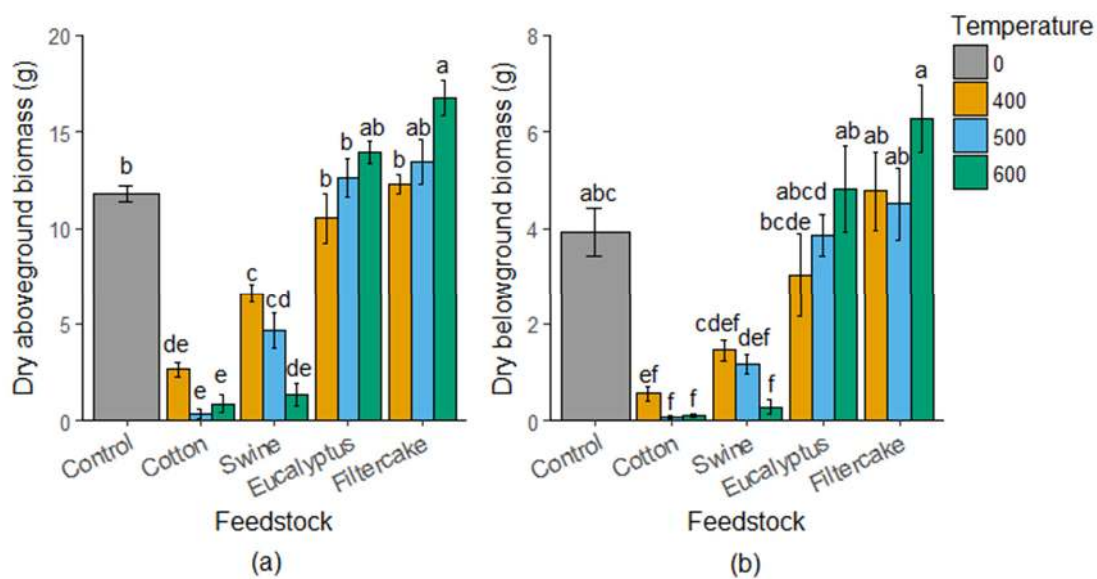


Figure 1. (a) Mean dry aboveground biomass (g); and (b) mean dry belowground biomass (g). Different letters represent significant differences between the biochar treatments including the control (Tukey test; $p < 0.05$).

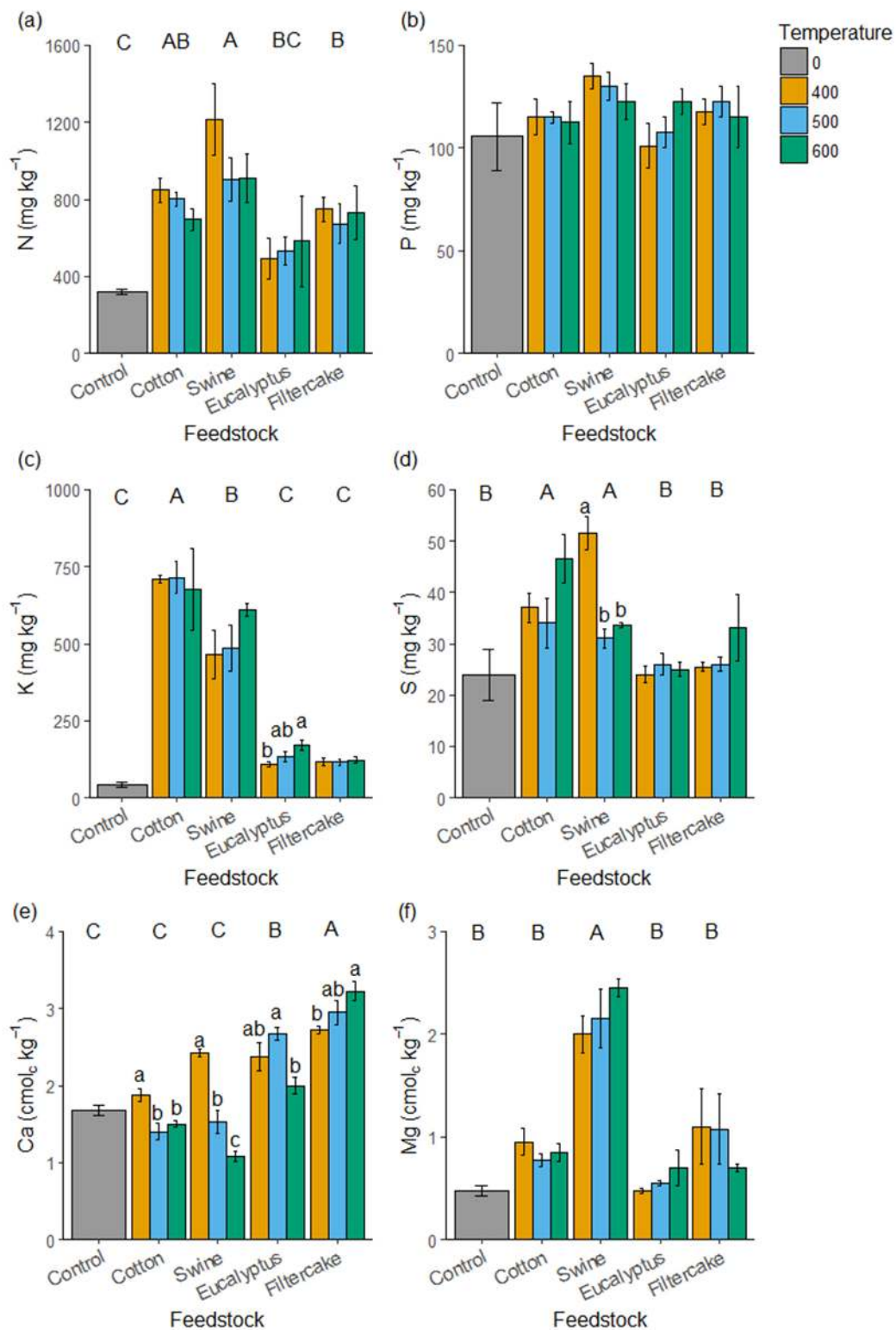


Figure 2. Mean concentration of (a) soil total nitrogen (N; mg kg^{-1}) and available soil macronutrients: (b) phosphorus (P; mg kg^{-1}); (c) potassium (K; mg kg^{-1}); (d) sulfur (S; mg kg^{-1}); (e) calcium (Ca; $\text{cmol}_c \text{ kg}^{-1}$); and (f) magnesium (Mg; $\text{cmol}_c \text{ kg}^{-1}$) in soils with different biochars. Capital letters indicate significant differences between the feedstocks and lowercase letters indicate significant differences between the temperatures for each feedstock (ANOVA, Tukey post-hoc test, $p < 0.05$); where absent, differences were not significant.

2.2. Water Content and EC Measurements

Weekly volumetric water content (θ) measurements showed that cotton and swine manure biochar treatments had significantly ($p < 0.05$) greater θ than eucalyptus, filtercake, and control treatments. Only swine manure biochar displayed significant differences between the temperatures, where θ of soils with swine manure biochar at 600 °C was greater than that at 500 °C (Figure 3a). EC measurements were similar, cotton biochar treatments having the highest mean EC, followed by swine manure biochar, and lastly eucalyptus, filtercake, and control treatments which were not different from each other. The differences between temperatures for swine manure and eucalyptus biochars were similar as observed for θ . Cotton and filtercake biochars, however, had significant differences, with EC in soils with cotton biochar at 600 °C significantly greater than that at 400 °C, while the opposite was observed for soils with filtercake biochars (Figure 3b). Osmotic potential (OP) determined from EC was lower in cotton and swine manure biochar treatments compared to the control and filtercake and eucalyptus biochars at higher temperatures. There were no significant differences ($p < 0.05$), however, between the temperatures for each feedstock (Table 1).

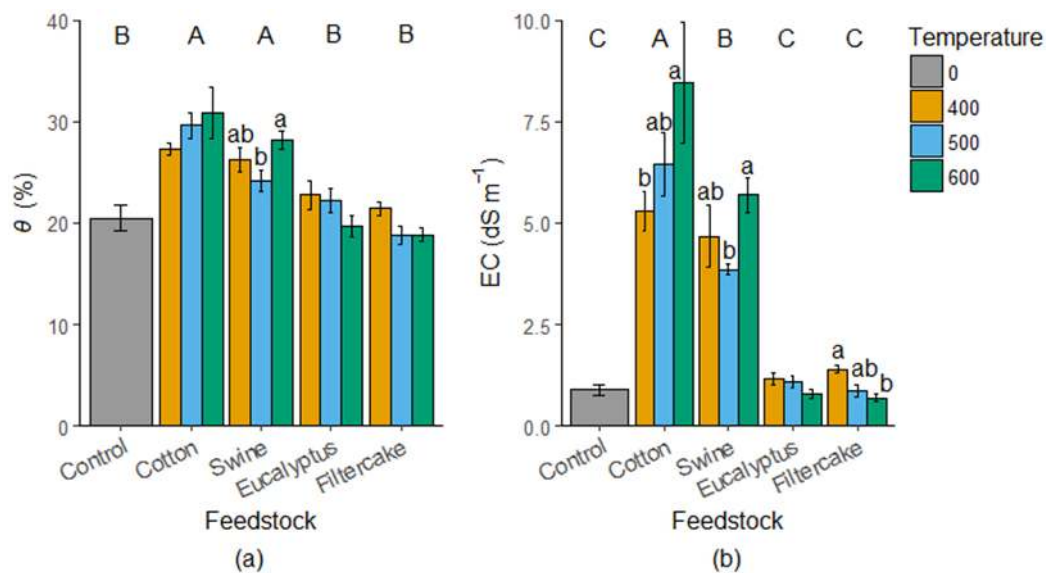


Figure 3. (a) Mean volumetric water content (θ ; %) and (b) electrical conductivity (EC; dS m^{-1}) over 6 weeks ($n = 5$). Capital letters represent significant differences between the feedstocks while lowercase letters represent significant differences between the temperatures (Tukey test; $p < 0.05$).

Table 1. Soil osmotic potential (OP) from each treatment. Lowercase letters indicate significant differences between the treatments ($n = 4$, Tukey test, $p < 0.05$).

Treatment	OP (−kPa)
Control	44.2 ± 6.4 d
Cotton400	264.7 ± 25.0 b
Cotton500	322.6 ± 39.8 ab
Cotton600	423.4 ± 74.2 a
Swine400	233.3 ± 38.0 b
Swine500	192.7 ± 6.2 bc
Swine600	285.3 ± 21.4 b
Eucalyptus400	57.9 ± 7.7 cd
Eucalyptus500	54.2 ± 8.2 d
Eucalyptus600	39.9 ± 5.9 d
Filtercake400	69.7 ± 4.7 cd
Filtercake500	43.5 ± 7.3 d
Filtercake600	34.5 ± 4.9 d

The correlation between EC and θ showed a positive relationship between the two parameters, with an R of 0.92 ($p < 0.001$). Plant biomass was also significantly and negatively correlated with both mean θ , EC, and AWC, as well as osmotic potential (OP) (Table 2), with biomass decreasing with increasing θ , EC, and AWC and with more negative OP.

Table 2. Pearson correlation coefficients (R) between final maize biomass and mean θ (%), mean EC (dS m^{-1}), available water content (AWC) (%), and OP ($-\text{kPa}$) over 6 weeks. *** = $p < 0.001$.

	θ (%)	EC (dS m^{-1})	AWC (%)	OP ($-\text{kPa}$)
Aboveground dry biomass	−0.88 ***	−0.88 ***	−0.71 ***	−0.88 ***
Belowground dry biomass	−0.83 ***	−0.80 ***	−0.62 ***	−0.80 ***

2.3. Water Retention Curves

Water retention curves determined from intact soil cores reflected mean θ for each treatment (Figure 4). Cotton biochar treatments had higher water retention, followed by swine manure, eucalyptus, and filtercake biochars, which were almost indistinguishable from each other. The control treatment had the lowest water retention. For AWC, cotton and swine manure biochar treatments had the highest levels and were significantly (Tukey test, $p < 0.05$) different from the control, but swine manure biochar did not differ from eucalyptus and filtercake biochars. The latter were also not significantly different from each other or the control, although they had higher mean AWC compared to the control. Only cotton biochar had significant differences between the temperatures, with AWC higher in soils with 600 °C cotton biochar than in soils with 500 °C biochar (Figure 5).

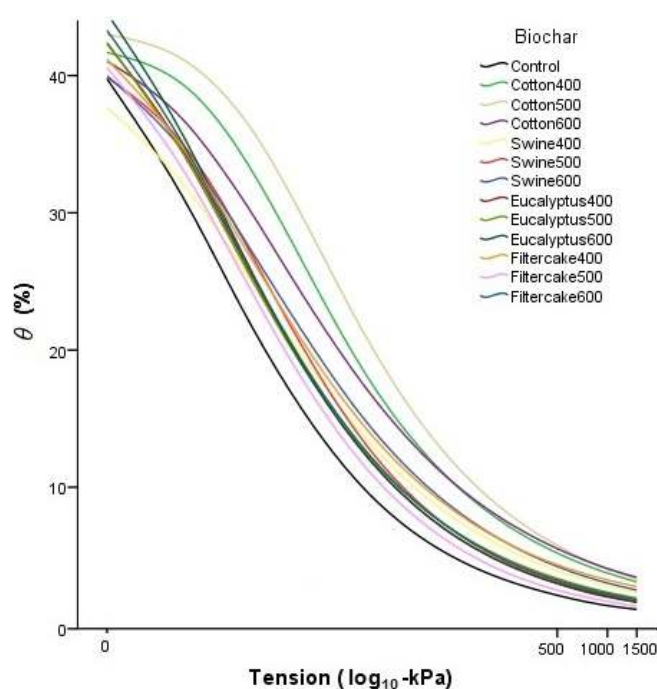


Figure 4. Water retention curves of intact soil cores ($n = 4$).

Final bulk density determined from intact soil cores showed no differences between the temperatures for each feedstock, and little difference between the feedstocks. Although most of the biochar treatments had lower mean bulk density than the control, only the bulk densities of soil with cotton biochar at 600 °C and eucalyptus biochar at 600 °C were significantly lower (Table 3).

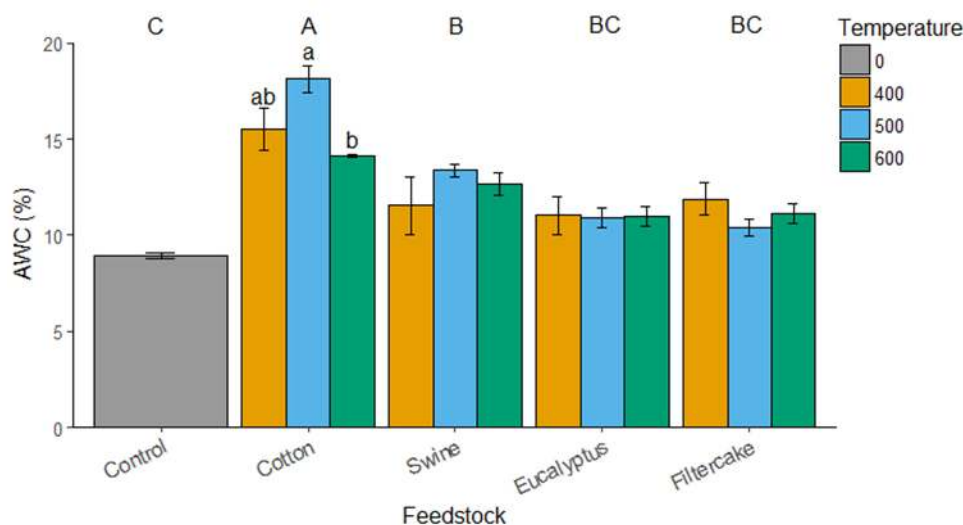


Figure 5. Mean available water content (%) determined from intact soil cores ($n = 4$). Capital letters represent significant differences between the feedstocks while lowercase letters represent significant differences between the temperatures (Tukey test; $p < 0.05$).

Table 3. Bulk density from intact soil cores. Lowercase letters indicate significant differences between the treatments ($n = 4$, Tukey test, $p < 0.05$).

Treatment	Bulk Density (g cm^{-3})
Control	1.38 ± 0.03 a
Cotton400	1.23 ± 0.05 abc
Cotton500	1.23 ± 0.03 abc
Cotton600	1.20 ± 0.04 bc
Swine400	1.30 ± 0.04 abc
Swine500	1.35 ± 0.03 ab
Swine600	1.30 ± 0.00 abc
Eucalyptus400	1.28 ± 0.05 abc
Eucalyptus500	1.23 ± 0.03 abc
Eucalyptus600	1.18 ± 0.03 c
Filtercake400	1.30 ± 0.04 abc
Filtercake500	1.33 ± 0.03 abc
Filtercake600	1.28 ± 0.03 abc

2.4. Particle Size Analysis

2.4.1. Biochar Characteristics

Particle size did not vary much between the biochars, except for the particle size of filtercake biochars, which was significantly lower than the others (Table 4). Between the temperatures of pyrolysis for each feedstock, there were no differences for cotton and swine manure, while filtercake at 400 °C had larger particle size than at 500 °C, and eucalyptus 400 °C and 600 °C larger than at 500 °C (Table 4). On examining the finest (D10) and coarsest (D90) parts of the grain size distribution [24] between feedstocks, filtercake biochar had the lowest D90, while there were no differences in D10. There were differences between the temperatures of pyrolysis for each feedstock for both D10 and D90, but no consistent trend. Correlation between biochar particle size and D90 showed a strong positive correlation ($R = 0.88$; $p < 0.001$). Particle size and total pore volume also showed a significant yet negative correlation ($R = -0.69$; $p < 0.001$), with total pore volume decreasing with increasing particle size.

Table 4. Porosity determined by BET-N₂ sorption ($n = 1$) and particle size and distribution of 12 biochars (4 feedstocks \times 3 temperatures of pyrolysis). Micropores are identified as pores <2 nm diameter, mesopores as pores between 2 nm and 50 nm diameter. For particle analysis, capital letters indicate significant differences between the feedstocks and lowercase letters between the temperatures of pyrolysis for each feedstock ($n = 3$; Tukey test, $p < 0.05$).

Biochar	Total Surface Area (m ² g ⁻¹)	Mesopore Area (m ² g ⁻¹)	Micropore Area (m ² g ⁻¹)	Total Pore Volume (cm ³ g ⁻¹)	Mesopore Volume (cm ³ g ⁻¹)	Micropore Volume (cm ³ g ⁻¹)	Particle Size (μm)	D10 (μm)	D90 (μm)
Cotton400 ^{*,†}	0.2	n/a	n/a	0.0017	n/a	n/a	A 888.2 ± 131.1 a	A 33.0 ± 7.3 ab	A 2790.5 ± 20.3 a
Cotton500	1.8	0.47	2.4	0.0056	0.0050	0.0012	757.2 ± 27.3 a	24.6 ± 1.3 b	2747.6 ± 24.7 a
Cotton600	1.9	0.53	2.9	0.0061	0.0055	0.0014	806.4 ± 53.8 a	53.7 ± 7.2 a	2715.9 ± 19.3 a
Swine manure400	7.2	4.1	0.7	0.0323	0.0289	0.0002	A 966.3 ± 94.6 a	A 210.0 ± 8.9 a	A 2733.8 ± 0.03 a
Swine manure500	24.9	9.6	9.7	0.0725	0.0596	0.0046	779.2 ± 51.9 a	61.0 ± 2.2 a	2706.6 ± 10.7 a
Swine manure600	36.9	10.1	15.4	0.0715	0.0524	0.0073	822.7 ± 25.2 a	143.2 ± 40.6 a	1606.7 ± 3.3 b
Eucalyptus400 [†]	0.3	n/a	2.3	0.0003	n/a	0.0011	A 860.6 ± 43.9 a	A 47.4 ± 7.9 a	A 2808.7 ± 10.0 a
Eucalyptus500	42.3	4.9	31.2	0.0520	0.0312	0.0151	415.6 ± 43.5 b	37.3 ± 3.4 a	476.9 ± 38.6 b
Eucalyptus600 ^{*,†}	132.0 ^{††}	n/a	n/a	0.0700	n/a	n/a	965.5 ± 71.8 a	45.5 ± 6.9 a	2824.3 ± 6.1 a
Filtercake400	13.5	10.3	0.4	0.0851	0.0787	0.0000	B 457.9 ± 7.3 a	A 42.4 ± 1.6 a	B 1241.6 ± 61.7 a
Filtercake500	25.0	14.6	5.1	0.1210	0.1095	0.0021	215.4 ± 37.9 b	31.1 ± 0.7 b	601.7 ± 156.8 a
Filtercake600	41.3	17.6	12.7	0.1314	0.1112	0.0059	363.2 ± 65.2 ab	38.4 ± 2.9 ab	947.9 ± 212.4 a

* Sample with poor N₂ absorption for which micropores were not detected and/or could not be measured. [†] No points within the specified BJH reporting interval. ^{††} Single point, rather than BET, surface area was calculated because of low N₂ absorption.

2.4.2. Biochar-Soil Mixtures

The sand aggregate size of soils with filtercake biochar was significantly greater ($p < 0.05$) than that of cotton and swine manure biochars, but not different from soil with eucalyptus biochar (Table 5). Sand aggregate size of soils with eucalyptus, cotton, and swine manure biochars did not differ between each other, and soils with all biochars had significantly greater ($p < 0.05$) sand aggregate sizes than the control (Table 5). Between the temperatures of pyrolysis for each feedstock, the sand particle size fraction in soils with cotton biochar at 600 °C was greater than at 400 °C and 500 °C; soils with swine manure biochar at 600 °C had greater sand particle size than at 500 °C, but neither differed from swine manure biochar at 400 °C. Soils with eucalyptus biochar at 400 °C had greater sand aggregate size than at 500 °C, but neither differed from soils with eucalyptus biochar at 600 °C. Lastly, sand particle size in soils with filtercake biochar at 600 °C was significantly greater than at 400 °C, but not greater than at 500 °C. Comparing silt-clay aggregate size between the treatments, there were no significant differences between the feedstocks or with the control, but there were differences between the temperatures of pyrolysis for each feedstock (Table 5). As with its sand aggregate size, cotton biochar at 600 °C contributed to greater silt-clay aggregate size than 400 °C and 500 °C. Soils with swine manure and filtercake biochars showed decreasing silt-clay aggregate size with increasing temperature of pyrolysis, and soils with eucalyptus at 400 °C had lower silt-clay aggregate size than at 500 °C and 600 °C (Table 5).

On examining the D10 and D90 of the grain size distribution, the fine sand (sand D10) did not differ between the feedstocks, whereas the coarse sand (sand D90) was greatest in soils with filtercake biochars and lowest in the control soil. Soils with cotton and filtercake biochars had similar silt + clay D10 and greater than the control, but silt + clay D90 did not vary. Comparing temperatures within the feedstocks, soils with cotton and filtercake biochars had higher sand D90 at 600 °C than at 400 °C, while silt + clay D90 was lower at 600 °C than at 400 °C for filtercake biochar, but higher at 600 °C for cotton biochar (Table 5).

Table 5. Aggregate size and distribution of biochar-soil samples. Capital letters before values indicate significant differences between the feedstocks and lowercase letters indicate significant differences between the temperatures of pyrolysis for each feedstock ($n = 3$; Tukey or Games-Howell tests, $p < 0.05$, where variances were unequal).

Treatments		Sand Aggregate Size (μm)		Silt + Clay Aggregate Size (μm)		Sand D10 (μm)		Sand D90 (μm)		Silt + Clay D10 (μm)		Silt + Clay D90 (μm)
Control	C	216.3 \pm 10.8	A	6.3 \pm 0.51	A	103.6 \pm 1.7	C	367.0 \pm 28.5	B	0.15 \pm 0.0	A	21.9 \pm 1.7
Cotton400	B	392.4 \pm 5.1 b	A	5.8 \pm 0.38 b	A	106.8 \pm 0.8 a	B	822.6 \pm 30.6 b	A	0.19 \pm 0.0 b	A	20.2 \pm 1.3 b
Cotton500		395.3 \pm 31.3 b		6.5 \pm 0.15 b		97.6 \pm 3.0 b		839.5 \pm 161.7 b		0.20 \pm 0.0 a		21.9 \pm 0.6 b
Cotton600		537.7 \pm 20.5 a		7.9 \pm 0.22 a		108.5 \pm 0.7 a		1694.8 \pm 76.4 a		0.19 \pm 0.0 b		26.1 \pm 0.7 a
Swine manure400	B	467.8 \pm 25.6 ab	A	7.1 \pm 0.43 a	A	106.6 \pm 0.7 b	B	1188.3 \pm 158.6 ab	AB	0.21 \pm 0.0 a	A	24.9 \pm 0.4 a
Swine manure500		345.1 \pm 5.6 b		4.8 \pm 0.15 b		104.2 \pm 2.0 b		636.0 \pm 11.7 b		0.14 \pm 0.0 b		17.4 \pm 1.5 b
Swine manure600		460.7 \pm 5.7 a		2.5 \pm 0.15 c		114.3 \pm 2.2 a		1240.1 \pm 24.6 a		0.13 \pm 0.0 b		8.8 \pm 1.5 c
Eucalyptus400	AB	604.8 \pm 64.5 a	A	3.5 \pm 0.1 b	A	115.1 \pm 0.9 a	AB	1629.9 \pm 201.1 a	B	0.16 \pm 0.0 a	A	11.2 \pm 1.4 b
Eucalyptus500		402.8 \pm 27.1 b		6.7 \pm 0.3 a		105.4 \pm 2.6 b		859.3 \pm 100.1 b		0.15 \pm 0.0 b		24.7 \pm 0.2 a
Eucalyptus600		450.8 \pm 31.5 ab		6.3 \pm 0.6 a		106.3 \pm 2.0 b		1179.2 \pm 166.6 ab		0.19 \pm 0.0 ab		20.0 \pm 0.7 a
Filtercake400	A	481.5 \pm 64.8 b	A	9.2 \pm 0.3 a	A	129.6 \pm 25.0 ab	A	1174.3 \pm 61.9 b	A	0.20 \pm 0.0 a	A	30.9 \pm 0.6 a
Filtercake500		775.0 \pm 4.4 ab		6.4 \pm 0.2 b		117.4 \pm 0.5 a		2589.0 \pm 103.6 a		0.20 \pm 0.0 a		22.8 \pm 0.6 b
Filtercake600		932.5 \pm 58.4 a		5.9 \pm 0.3 c		100.0 \pm 2.8 b		2829.3 \pm 8.9 a		0.19 \pm 0.0 a		21.6 \pm 1.2 b

3. Discussion

3.1. Effect of Biochar Feedstock on Plant Biomass

The results of the greenhouse experiment suggest that water retention and nutrient levels, as suggested by EC, influenced plant biomass in biochar treatments. Filtercake and eucalyptus biochars did not significantly alter mean θ , EC, plant AWC, or OP in soils compared to the control, but maintained high maize biomass; filtercake biochar at 600 °C even leading to a 33% increase in maize biomass compared to the control (Figure 1). In contrast, the high θ , EC, and AWC, and lower OP in soils with cotton and swine manure biochars led to lower maize biomass, as noted by the negative correlations (Table 1) and visually evident (Figure S3). When added to the soil, biochar has the potential to increase pores in the 30 to 0.3 nm diameter range [25], but the pore size range for plant AWC is 0.2 to 30 μm diameter [19]. In soils with cotton and swine manure biochars, the biochars may have increased soil porosity in the AWC pore range, leading to high AWC and nutrient content, but the water was immobile and thus unavailable for plant uptake or solute transport [23]. In addition, soils with cotton and swine manure biochars had high available K and S levels, as well as high total N and available Mg in soils with swine manure biochars (Figure 2). EC can indicate salinity, which refers to “the presence of major dissolved inorganic soil solutes in the soil aqueous phase” [26] (p. 17), including nutrients (or salinity ions) such as Na^+ , K^+ , Ca^{+2} , Mg^{+2} , SO_4^{-2} and NO_3^- . EC can be used then to determine crop yield potential. The large presence of dissolved ions, however, can affect the soil osmotic potential, reducing plants’ ability to uptake water, as well as disrupting plants’ nutritional balance [26]. Biochar macropores in particular can discourage root hair growth into them because they can be saturated with immobile water with high concentrations of salinity ions or phytotoxic organic compounds [27]. Thus despite having high θ , EC, and AWC, the lower OP in soils with cotton and swine manure biochars, likely related to high K levels, may not have allowed maize plants to uptake water and nutrients, limiting their growth. Rajkovich et al. [28] similarly observed low maize growth at high biochar application rates (7% *w/w*) due to high salinity caused by Na. Post-treatment such as rinsing the biochar or mixing with other biochar types, as well as appropriate application rates, could help reduce dissolved ion levels [28]. In contrast, OP in soils with eucalyptus and filtercake biochars, particularly at higher temperatures, had high OP, allowing for free movement of water and soil aeration. In addition, filtercake biochars contributed to high soil Ca levels, which were positively correlated with higher dry aboveground biomass. Major et al. [29] noted that maize growth declined in a savannah Oxisol with low Ca and Mg levels; lower soil Ca levels in cotton and swine manure biochar treatments may similarly have also limited maize growth in our study. These differences in maize biomass may thus be related to nutrient content, but more likely to salinity levels affecting net osmotic potential on total soil water potential.

3.2. Biochar Contribution to Soil Water Retention

Water retention and nutrient absorption by biochar is related to its high porosity [25,30,31]. Because of its high porosity, biochar has been shown to increase AWC, as well as plant available water at the permanent wilting point [9], mainly due to the high amount of micropores [19]. Biochar’s porosity is determined both by the feedstock (which contributes mostly to macropores) and to the temperature of pyrolysis (which contributes to micropores and nanopores) [18,32]. While soil macropores (>80 μm) allow for water flow and soil mesopores (between 30 μm and 80 μm) allow gradient movement, soil micropores (<30 μm) retain water [30]. In biochar, residual macropores (1 to 100 μm) are formed from plant cellular structure and are believed to contribute to biochar pore volume, while pyrogenic nanopores (<2 nm) are formed during pyrolysis and contribute mostly to biochar surface area [21]. In sandy soils, biochar can behave like a clay-size particle, holding large amounts of water which may be accessible to plants. High porosity is related to high surface area, which allows for greater nutrient adsorption. Biochar surface area usually increases with temperature of pyrolysis [30], as was the case

for our biochars (Table 4), where total surface area and micropore volume increased as temperatures increased from 400 to 600 °C.

When our biochars were mixed with soil, however, the effect of biochar temperature of pyrolysis on water and nutrient retention was less than differences due to feedstocks. This is in agreement with Jeffery et al. [33], who did not observe either a biochar effect on water retention nor differences between the biochars at 400 °C and 600 °C, despite the biochar 600 °C having higher porosity. Although temperature of pyrolysis is considered the most important factor determining physical changes of biochar, feedstock type determines the temperature range under which changes occur [15]. Typically, the pore structure of a biochar resembles the cellular structure of the feedstock in wood or plant-based biochars. The organic and inorganic components (e.g., ash) can affect biochar structure as temperatures of pyrolysis increase, increasing decomposition and/or reacting with the C lattice structure [34]. Yet, on examining scanning electron microscopy (SEM) images of our 12 biochars (Figures S4 and S5), pores do not appear to differ between the temperatures of pyrolysis (400, 500 and 600 °C) for each feedstock. Comparing the feedstocks, however, micropores in cotton and eucalyptus biochars appear stacked and longitudinal, while micropores in swine manure biochar appear irregularly, like pores in a sponge. Unlike the other biochars, micropores in filtercake biochar are barely visible, corroborated by its low micropore volume (Table 4). Eykelbosh et al. [35] likewise observed large irregular macropores with few micropores in SEM images of their filtercake biochar produced at 575 °C.

Filtercake biochar's low micropore volume, but high total pore and mesopore volumes and surface areas, suggests that it contained more meso- and macropores that allowed for sufficient drainage in the soil, and therefore θ and AWC in soils with filtercake biochar did not differ from the control. Eucalyptus biochar had high total surface area, but low total pore volume. Biochars made from wood often have large macropores (~10 μm) [36], but the low total pore volume of the eucalyptus biochars may suggest it contained smaller macropores or mesopores. Lee et al. [37] observed high surface area for palm nut kernel shell biochar heated to 500 °C due to the presence of mesopores. Although mesopores for only eucalyptus biochar at 500 °C were measurable in our analysis, more small mesopores, rather than micropores, in eucalyptus biochar could have also promoted drainage so that θ in soils with eucalyptus biochar did not differ from filtercake biochar treatments or the control.

The high θ in soils with cotton and swine manure biochars would suggest that these biochars had high porosity and surface area that contributed to high soil water retention. Swine manure biochars had high total surface area similar to filtercake biochars, but they contained lower mesopore surface area and volume and higher micropore surface area and volume than filtercake biochars. Less mesopores and more micropores may have contributed to greater water retention in soils mixed with swine manure biochars. The cotton biochars, however, surprisingly had the lowest total surface area and micropore volume of all the biochar feedstocks, although its micropore surface area was greater than its total surface and mesopore area (Table 4). In the literature it has been noted that the hydrologic properties of biochars cannot be entirely determined before adding it to the soil. A biochar can have different effects on the soil hydrology depending on the soil, so that the hydrology of the biochar-soil mixture is not directly related to the hydrology behaviour of biochar alone [23]. Kinney et al. [38] observed that the field capacity of different pure biochars did not lead to similar field capacity in biochar-soil mixtures. This appears to be the case for cotton biochar in our study, which contributed to high soil water retention, despite the cotton biochars alone having overall lower porosity and surface area than the other biochars (Table 4). However, in the presence of plants, it was evident that, despite the high θ and AWC, maize plants were unable to uptake water at the same soil moisture potential due to lower OP. Nevertheless, if applied at a more suitable rate, cotton biochar could potentially improve soil θ and AWC without significantly lowering OP.

3.3. Biochar Particle Size and Distribution

Particle size analysis of the biochars showed filtercake biochar had significantly lower mean particle size than the other biochars, which did not differ from each other (Table 4). In addition,

filtercake biochar had lower D90 than the other biochars. The negative correlation between total pore volume and particle size of the biochars suggests that filtercake biochar's high total pore volume was influenced by its low particle size and low coarse fraction distribution. Cotton biochars had low total pore volume, but large particle size. Swine manure and eucalyptus biochars also had large particle sizes compared to filtercake biochar, but higher total pore volume than cotton biochar (Table 4). Biochars can alter soil water dynamics by changing the size, shape and amount of pores between soil particles [23]. For cotton biochar in particular, its large particle size may have reduced some or all of these pore characteristics in the soil.

When mixed in soil, the biochars led to significantly greater sand aggregate sizes compared to the control, with soils with filtercake biochars having the highest mean sand aggregate size (Table 5), despite the biochar alone having a low coarse fraction (Table 4). Larger biochar particles might remain closer to the soil surface, while smaller particle size could allow the biochar to move further down the soil profile [39]. Biochar added in one layer has been observed to reduce water loss in a sandy soil compared to biochar mixed uniformly, as the biochar layer can slow down water flow [40]. Soils with filtercake biochars also had high sand D90 and silt + clay D10, significantly greater than the control (Table 5), which may have contributed to greater heterogeneity in grain size distribution in the soil mixture. In addition, Crawford et al. [41] observed that soil porosity was lowest when the fine-grained particle volume filled the pore space of the coarse-grained fraction. Filtercake biochar may have somewhat reduced soil porosity if its silt + clay fraction matched the soil pore space, allowing soil water to flow freely rather than retaining it. The greater heterogeneous distribution, high coarse sand and fine silt + clay fractions, and high sand aggregate sizes in soils with filtercake biochar (especially at 600 °C compared to at 400 °C) may have all allowed for water to flow and drain more freely, thus providing low water retention, but without impacting plant growth. Eucalyptus biochar likewise contributed to a high sand D90 and silt + clay D10 similar to the control, also leading to low water retention, although both biochars nevertheless had higher mean AWC than the control. Furthermore, the high Ca:Mg ratios in filtercake and eucalyptus biochar treatments suggest better soil structure, with higher ratios related to improved water infiltration [42]. Soils with greater Ca than Mg can improve soil aggregation by increasing flocculation, leading to less surface sealing and higher infiltration rates [43].

In contrast, soils with cotton and swine manure biochars had silt + clay D10 similar to soils with filtercake biochar, but both had lower sand D90. Separating miscanthus and wheat biochars into three particle size fractions (0–500 µm, 500–1000 µm, and 1000–2000 µm) and mixing into a loamy sand, Glab et al. [22] observed higher field capacity and AWC in soils with the 0–500 µm biochar fraction applied at 4% *w/w*. In the present study, the high fine silt + clay fraction in soils with cotton and swine manure biochars could have also led to their higher mean water retention and AWC (Figures 3 and 4) compared to the filtercake and eucalyptus biochars, as well as the control. Furthermore, larger biochar particles were visible on the soil surfaces of pots with cotton and eucalyptus biochars (e.g., the 600 °C biochars, Figure S6). In the case of cotton biochar, the formation of biochar layers within the soil combined with its low heterogeneous grain size distribution and high contribution to the fine silt + clay fraction may have exacerbated soil water retention.

Both grain size distribution and particle size of biochars can be used to predict their impact on saturated hydraulic conductivity (K_s). Lim et al. [44], using their model of large (>1 mm) and small particle size (<1 mm), observed that biochars with large particle size significantly decreased soil K_s in their study, consistent with similar studies they reviewed. This effect was particularly noticeable in coarse and fine sandy soils. Barnes et al. [17] also recorded decreased K_s in biochar-sandy soil mixtures, perhaps due to increased tortuosity in the interstitial space between biochar and sand particles. Using Lim et al.'s [44] model, we can predict that K_s in our biochar-sandy soil mixtures decreased compared to the control soil, since all biochars had significantly greater sand particle sizes and D90 than the control. A preliminary assessment of K_s in our biochar-sandy soil mixtures using a HYPROP® (UMS GmbH, Munich, Germany) [45,46] corroborated this prediction (Table S1). Values were lowest in soils with swine manure biochars and with cotton biochar at 600 °C and eucalyptus at 400 °C, while soils

with filtercake biochars had slightly higher K_s , but still lower than the control (Table S1), consistent with its high contribution to the soil sand fraction (Table 5). Hydraulic conductivity, K , in control soils was lower than K in biochar-soil mixtures from pF 2 to past the permanent wilting point (Figure S7). This suggests that in control soils water drained more easily and moved more quickly through the soil when saturated compared to in soils with biochar. More testing, however, is required to confirm the influence of these biochars on K_s and K .

4. Materials and Methods

4.1. Soil Collection and Biochar Production

Soils from the top 0–20 cm layer were collected from an agricultural field located within the farm Fazenda Água Azul (15°13'55.2'' S, 54°57'43.4'' W) managed by the agribusiness Grupo Bom Futuro, 178 km northwest of the state capital of Cuiabá in Mato Grosso, Brazil, an area within the Cerrado biome. The soil collected was classified as an Arenosol (FAO soil classification), with a sandy texture (91% sand, 4% silt, 5% clay). Carbon (C) and nitrogen (N) levels in the soil were 0.7% C and 0.08% N as determined by elemental analysis (628 Series CHN Analyzer, LECO Corp., St. Joseph, MI, USA). The average pH_{water} was 5.8 and average CEC was 5.3 $\text{cmol}_c \text{ kg}^{-1}$, with a bulk density of 1.6 g cm^{-3} . Over the last 10 years, the crops sown on the study site included soybean, sorghum, maize, and cotton, with the latter two crops grown in rotation with soy for the last three years [47]. Twelve biochars were commercially produced (SPPT Ltd., Mogi Morim, São Paulo, Brazil) from four feedstock materials: cotton husks, eucalyptus sawmill residue, sugarcane filtercake, and swine manure, slow-pyrolyzed at three temperatures (400 °C, 500 °C, 600 °C). These were subsequently ground and sieved to <2 mm.

4.2. Experimental Design

Carried out at the Federal University of Mato Grosso, Cuiabá campus, the greenhouse experiment consisted of 9 L volume pots with one hole drilled at the bottom filled with 8 kg of an Arenosol. Twelve biochars (4 biochar feedstocks \times 3 temperatures of pyrolysis) were applied to pots at 5% soil dry weight, mixed and compacted by hand, making a total of 52 pots (12 biochars \times 4 replicates plus 4 unamended soil controls). A high biochar application rate (equivalent to 80 tonnes ha^{-1}) was used to ensure a biochar effect was detected. The pots were divided into 4 blocks, with each block running north-south along a greenhouse bench, with a replicate of each treatment (biochar amended soil) plus a control (unamended soil) randomly assigned to locations within each block. Fertilizer, 2.5 g NPK+S (12-46-0 + 7), was added after 1 week and four maize seeds were planted in each pot. Pots were watered three times a week to maintain soil moisture at 60% of field capacity for 45 days. Crushed KCl (2.5 g) and diluted urea (2.0 g in 50 mL water) were added 20 days after planting, followed by a second diluted urea application of 1.3 g 7 days later. Volumetric water content (θ) and EC were directly measured and recorded once a week the day after a watering event using a GS3 sensor (Decagon Devices, Inc., Pullman, WA, USA). At the end of the experiment, above and belowground maize biomass was collected, weighed fresh, then dried at 60 °C for 48 h and reweighed. Soil samples were collected and analyzed for macronutrient (P, K^+ , Ca^{2+} , Mg^{2+} , and S) availability according to the standard soil methodologies used by the Empresa Brasileira de Pesquisa Agropecuária [48] as described in Eykelbosh et al. [35]. Soil total N was analyzed on a CHN Analyzer (628 Series, LECO Corp., St. Joseph, MI, USA).

4.3. Water Retention Curves

After biomass collections, intact soil cores (100 cm^3) were taken from each pot, resulting in 52 cores to be used in the laboratory. A fine mesh was placed at the bottom of each soil core and the cores placed in a pan of water to saturate for 24 h until reaching equilibrium before placing them in a tension table to determine θ at 0, 2, 4, 6, 8, and 10 kPa [49]. Afterwards they were transferred to pressure chambers to determine θ at 33 and 100 kPa. The samples were kept at each matric potential for one week then

weighed before moving to the next matric potential. θ at 500, 1000, and 1500 kPa were determined using the WP4C Dewpoint Potentiometer (Decagon Devices, Inc., Pullman, WA, USA) [50], as described by Eykelbosh et al. [35]. θ ($\text{cm}^3 \text{cm}^{-3}$) for all matric potential points (0–1500 kPa) was then entered into the Soil Water Retention Curve software (SWRC, version 2.0, University of São Paulo, São Paulo, Brazil) [51] to adjust the soil water retention, θ , of each replicate using the unimodal constrained model of van Genuchten [52]:

$$\theta = \theta_r + \frac{\theta_s - \theta_r}{[1 + (\alpha \psi_m)^n]^m}$$

where $m = 1 - 1/n$ [53], θ is volumetric water content, ψ_m is matric potential, θ_r is residual θ , θ_s is saturated θ , and n and α are adjusted parameters. The results were then used to obtain AWC and water retention curves for each treatment ($n = 4$). AWC (%) was calculated as θ at 33 kPa (field capacity) minus θ at 1500 kPa (permanent wilting point). Final bulk density was also determined from intact soil cores. Osmotic potential (OP) was calculated using the universal equation of OP to EC [54], $OP(\text{kPa}) \approx -50 * EC(\text{dS m}^{-1})$.

4.4. Biochar Particle Size and Porosity Analysis

For particle size analysis in the laboratory, the soil samples collected at the end of the experiment were separated into sand ($>53 \mu\text{m}$) and silt + clay ($<53 \mu\text{m}$) fractions following the fractionation method by EMBRAPA [55]. Briefly, sodium hydroxide (NaOH) and distilled water were added to 10 g of air-dried soil and shaken overnight. After this time, sand and silt + clay fractions were separated using a $53 \mu\text{m}$ sieve. Once separated, an aliquot of the silt + clay fraction in suspension was placed in a laser diffraction particle size analyzer (LA 950, Horiba Scientific, Edison, NJ, USA) to determine biochar-soil aggregate particle size. Afterwards, biochar-soil aggregate size of the oven-dried sand fraction was measured. Particle size of each biochar alone ($<2 \text{mm}$) was determined directly by the analyzer.

The Brunauer-Emmett-Teller (BET) total surface area [56], total pore volume, Barrett-Joyner-Halenda (BJH) mesopore surface area and volume [57], deBoer t-plot micropore surface area, and micropore volume of each biochar were determined from automated gas sorptometry with N_2 performed by an ASAP 2020 Plus Physisorption Analyzer (Micromeritics, Norcross, GA, USA). Results of the physical characterization of the biochars are shown in Table 4.

4.5. Statistical Analyses

The effects of biochar treatments on plant biomass, θ , AWC, EC, OP, bulk density, and particle size were determined by univariate analysis of variance (ANOVA) and multivariate (MANOVA) for grain size distribution (D10 and D90), using IBM[®] SPSS[®] Statistics software (version 23, SPSS, Inc., Chicago, IL, USA). Where treatments were significant, a post-hoc Tukey test ($p < 0.05$) was used to compare means, and a post-hoc Games-Howell ($p < 0.05$) test when variances were unequal as in the case of particle size and distribution. Pearson correlations and linear regressions were performed between plant biomass and soil macronutrients, mean θ , EC, AWC, and OP as well as between mean θ and EC and OP. Values presented in graphs are means and ± 1 standard errors.

5. Conclusions

Overall, the agricultural waste biochars assessed in this study showed potential for increasing water retention when mixed with an Arenosol, compared to the soil alone, and may decrease K_s . Analysis of the porosity, surface areas, and SEMs of the biochars did not always coincide with the effect of the biochars mixed with soil, especially when comparing temperatures of pyrolysis. Yet, the properties of the biochars cannot always predict its effect in the soil, as many factors come into play including soil and biochar particle size and the presence of plants. This was especially evident in cotton and swine manure biochar treatments which showed potential to increase soil water retention, plant AWC, and nutrient content more than unamended soils, but nevertheless had a negative effect

on maize biomass, likely due to lower OP preventing plants from benefiting from the high AWC. Therefore our hypothesis that higher temperature biochars would lead to higher water retention and EC, thereby increasing maize biomass, was only partly correct, as cotton and swine manure biochars at 600 °C had the highest mean θ and EC, but the lowest maize biomass. The high θ was probably related to their contribution to the fine silt + clay fraction in the soil, whereas filtercake biochar treatments, which had the highest mean maize biomass, contributed most to the soil coarse sand fraction. Both eucalyptus and filtercake biochars, particularly at 600 °C, may have contributed more to soil aeration and water infiltration, allowing for maize to grow well. In summary, our study showed that feedstock properties and application rates are important factors to determine when adding biochar to soil, in order to prevent excessive water and/or salinity that may negatively impact plant growth. To our knowledge, this is the first study to show an environmentally beneficial use of the waste biomass, filtercake. Both filtercake and eucalyptus biochars show potential for maintaining or even increasing plant growth, while applying cotton and swine manure biochars at lower levels may contribute to increased soil water resilience of Cerrado Arenosols. If applied at a suitable rate so as not to cause excessive salinity, these biochars could make a significant contribution to crop production in times of drought.

Supplementary Materials: The following are available online at www.mdpi.com/2073-4395/7/03/49/s1, Figure S1: Correlations between dry aboveground biomass (g) and (a) potassium (K; mg kg⁻¹) and (b) calcium (Ca; cmol_c kg⁻¹), Figure S2: Mean Ca:Mg ratios ($n = 4$), Figure S3: Maize plants in soils with 12 different biochars: cotton, swine manure, eucalyptus, and filtercake biochars at 400, 500, and 600 °C, Figure S4: SEM images of cotton and swine manure biochars at 400, 500, and 600 °C at 400× magnification, Figure S5: SEM images of eucalyptus and filtercake biochars at 400, 500, and 600 °C at 400× magnification, Figure S6: Maize plants in soils with cotton, swine manure, eucalyptus, and filtercake biochars at 600 °C in the early stages of the experiment, Figure S7: Hydraulic conductivity curves for packed biochar-soil mixtures, low and high temperatures of pyrolysis (HYPROP data; $n = 1$), Table S1: Parameter values for water content (θ) and K_s and fit quality of the model measured by root mean square error (RMSE)^a for each biochar-soil mixture treatment (HYPROP data).

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