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Low-Temperature Slow Pyrolysis: Exploring Biomass-Specific Biochar Characteristics and Potential for Soil Applications

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Abstract: The pyrolysis process of residues has emerged as a sustainable method for managing organic waste, producing biochars that offer significant benefits for agriculture and the environment. These benefits depend on the properties of the raw biomass and the pyrolysis conditions, such as washing and drying. This study investigated biochar production through slow pyrolysis at 300 °C, using eight biomass types, four being plant residues (PBR)—sugarcane bagasse, filter cake, sawdust, and stranded algae—and four non-plant-based residues (NPBR)—poultry litter, sheep manure, layer chicken manure, and sewage sludge. The physicochemical properties assessed included yield, carbon (C) and nitrogen (N) content, electrical conductivity, pH, macro- and micronutrients, and potentially toxic metals. Pyrolysis generally increased pH and concentrated C, N, phosphorus (P), and other nutrients while reducing electrical conductivity, C/N ratio, potassium (K), and sulfur (S) contents. The increases in the pH of the biochars in relation to the respective biomasses were between 0.3 and 1.9, with the greatest differences observed for the NPBR biochars. Biochars from sugarcane bagasse and sawdust exhibited high C content (74.57–77.67%), highlighting their potential use for C sequestration. Filter cake biochar excelled in P $(14.28 \text{ g kg}^{-1})$ and micronutrients, while algae biochar showed elevated N, calcium (Ca), and boron (B) levels. NPBR biochars were rich in N (2.28–3.67%) and P (20.7–43.4 g kg⁻¹), making them ideal fertilizers. Although sewage sludge biochar contained higher levels of potentially toxic metals, these remained within regulatory limits. This research highlights variations in the composition of biochars depending on the characteristics of the original biomass and the pyrolysis process, to contribute to the production of customized biochars for the purposes of their application in the soil. Biochars derived from exclusively plant biomasses showed important aspects related to the recovery of carbon from biomass and can be preferred as biochar used to sequester carbon in the soil. On the other hand, biochars obtained from residues with some animal contributions are more enriched in nutrients and should be directed to the management of soil fertility.

Keywords: carbon sequestration; fertilizer; organic waste management; soil amendment; toxic metal regulation



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Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/ licenses/by/4.0/). Modern agriculture is heavily reliant on soluble mineral fertilizers, which, together with other technologies, have driven the "Green Revolution" since the mid-20th century, significantly boosting global productivity and ensuring food security [1]. The three primary nutrients supplied through fertilizers are N, obtained by industrial methods, the P and K, that are extracted from geological sources, such as phosphate rock and potash deposits [1–4].

N fertilizer production uses a huge amount of energy in its process, relying on fossil sources and emitting significant amounts of C into the atmosphere, exacerbating global climate change. Due to low fertilization efficiency, N is lost to other compartments beyond the soil-plant system, negatively affecting human well-being, biodiversity, and ecological balance [5,6]. For P and K, extraction also requires significant energy and resources, and their overuse or mismanagement in soluble mineral fertilizers can lead to environmental degradation, such as eutrophication of water bodies from P runoff [7]. Additionally, with growing global demand for food and increasing pressure on these limited reserves, ensuring their sustainable use through efficient management practices, recycling, and the development of alternative sources is critical for maintaining long-term agricultural productivity and food security [2].

One way to reduce the use of soluble mineral fertilizers and minimize their negative environmental impacts is by recycling waste in agriculture, while maintaining competitive productivity and ensuring food security [4]. Furthermore, recycling nutrients from agricultural waste is also strategic for Brazil, as approximately 80% of all N-P-K applied is imported. This high dependence on imported fertilizers weakens the agricultural sector and makes it highly susceptible to geopolitical issues [8,9].

Organic waste can be used in the soil as a conditioner and/or nutrient provider for plants [10]. Technical standards need to be followed to ensure its effective, safe, and appropriate usage, including pretreatment methods that partially stabilize organic substances and remove potential pathogens and organic pollutants [11]. A viable option is pyrolysis to generate biochar, a C-rich substance created by pyrolyzing biomass in an oxygen-restricted environment. In pyrolysis, the organic compounds in biomass, including lignin and cellulose, undergo thermochemical changes in their structures, forming aromatic rings and providing greater stability. In addition, the high temperatures and duration of the pyrolysis process can eradicate pathogens, bacterial residues, and pesticides [11,12], resulting in materials that are safe for agricultural purposes.

These aromatic compounds in biochar are significantly more resistant to microbial decomposition compared to the original biomass, due to their condensed and dense structure, which hinders enzymatic and chemical access [13]. As a result, biochar can persist in the soil for decades to centuries, contributing to long-term C sequestration [14] and reducing net greenhouse gas emissions [15]. When incorporated into the soil, biochar also enhances soil structure, water retention, nutrient availability, and microbial activity, creating a more favorable environment for plant growth. This dual role of biochar in improving soil health and sequestering atmospheric C underscores its importance as a sustainable agricultural amendment and a key strategy for climate change mitigation [14,16].

Thermal decomposition temperature is crucial for defining biochar's chemical composition, stability, and adsorption capacity. Biochars pyrolyzed at low temperatures (up to 400 °C) offer distinct and advantageous characteristics for agronomic and environmental purposes. One notable aspect is the higher C yield in pyrolysis, with minimal losses during the process and higher concentrations in biochar [17]. These biochars exhibit higher organic C content, indicating reduced C loss during pyrolysis, which is vital for enhancing physical properties like water retention, chemical properties like cation exchange capacity (CEC) [18–20], and biological properties like microbial activity [21]. These characteristics allow managed and prolonged nutrient release in the soil while improving the soil's nutrient retention ability, decreasing dependence on mineral fertilizers [22,23]. Its neutral or alkaline pH also provides additional benefits, especially in acidic soils, improving nutrient availability and promoting a healthier root environment for plants [24]. Conversely, biochars produced at lower temperatures have lower aromaticity compared to those made at higher temperatures, making them less recalcitrant in the environment [25]. For addressing climate challenges, both the C content in biochar and its recalcitrance in the environment are critical. The Intergovernmental Panel on Climate Change (IPCC) suggests a value of 70% for biochar C stability in soil for over 100 years, while other estimates and models vary between 60% and 90%, depending on biochar characteristics, pyrolysis temperature, and ambient temperature [26–28].

The type of biomass used also determines biochar's chemical properties, stability, and functionality [29]. Non-plant-based residues (NPBR), such as animal waste or sewage sludge, are composed of proteins, amino acids, other organic compounds, and higher amounts of ash and inorganic materials, resulting in higher CEC and nutrient availability for plants [30,31]. Despite the high N concentration, they have low availability and should be used mainly as soil conditioners or enriched with mineral sources to formulate organomineral fertilizers [32]. Plant-based residues (PBR), such as agricultural residues, wood, and straw, have a lignocellulosic base, with cellulose, hemicellulose and lignin composing most of the biomass, resulting in biochars rich in stable aromatic compounds, ideal for C sequestration and enhancing soil physical properties like water retention [33]. Additionally, NPBR often requires pre-treatment to reduce contaminants such as heavy metals and pathogens, whereas PBR, typically cleaner, are better suited for environmental applications prioritizing C stability [30]. The diversity of biomasses and their characteristics highlight how careful selection can optimize biochar for specific uses, such as soil improvement and C sequestration [31,34].

Thus, the aim of this study was to assess the physicochemical properties of biochars generated by slow pyrolysis at low temperatures from different biomass sources, recognizing their possible applications for agricultural and environmental uses.

2. Materials and Methods

2.1. Origin of Residues and Preparation

The PBR were obtained from the state of São Paulo, Brazil, except for arribadas algae (AA), consisting of tropical macroalgae (*Gracilaria* spp. and *Hypnea* spp.), collected on the coast of Bahia state, Brazil. Sugarcane bagasse (SB) and filter cake (FC) were sourced from an ethanol and sugar mill in Usina Lins, in the municipality of Lins. Sawdust (SD), composed of residues from the sawing of pine (*Pinus* L.) and Cupiúba (*Goupia glabra* Aubl.), was obtained from Madeireira M.A.P.A., a sawmill in the municipality of Jaguariúna, SP, Brazil. Sewage sludge (SS), composted and dried, was obtained from a wastewater treatment plant in the municipality of Lins, using anaerobic lagoon systems. Chicken manure (CM), poultry litter (PL), and sheep bedding (OL) were obtained from research institutions in the zootechnics field located in the municipalities of Nova Odessa and Piracicaba, SP, Brazil. All residues were air-dried to constant weight, then grounded (TRF3000 Super Trapp®, Jaraguá do Sul, Brazil) and sieved through a 2 mm mesh. Biomasses were named BM-XX, where the last two characters refer to the raw material used for their production.

2.2. Biochar Production and Evaluations

The biomasses were maintained in an oven at 60 °C for four days, subsequently put into a sealed crucible for pyrolysis in a muffle furnace with limited oxygen availability. Biochars were generated through slow pyrolysis at 300 °C, with a heating rate of

 $1.5 \,^{\circ}$ C min⁻¹ until reaching the final temperature, held for 60 min. Containers and muffle were kept sealed until the system cooled to room temperature. The 300 $^{\circ}$ C temperature was selected to achieve higher mass yield and maintain favorable chemical characteristics like CEC and nutrient presence. After pyrolysis and cooling, the biochars were washed multiple times with deionized water, followed by drying at 60 $^{\circ}$ C in a forced-air oven. Biochars were designated BC-XX, where the last two characters refer to the raw material used.

Analysis in triplicate were conducted on the biomasses and biochars to evaluate changes in both physical properties and chemical composition caused by biochar production. Parameters evaluated included moisture (at 103 °C) and ash content (at 800 °C) using gravimetry [35]; electrical conductivity in an aqueous extract (1:10 mass-to-volume ratio); pH in 0.01 mol L⁻¹ CaCl₂ solution by potentiometry (EBC, 2022); total C and N contents using a CN analyzer [35–37]; nitrate and ammonium contents by the Kjeldahl method [38,39]; organic C by oxidation with potassium dichromate and subsequent titration with ammonium ferrous sulfate [37]; macronutrient, micronutrient, and potentially toxic metal (PTM) contents extracted following the SW-846 method 3051a protocol and determined by inductively coupled plasma optical emission spectrometry [40]. Analyses were conducted at the Residues and Fertilizers Laboratory of the Agronomic Institute in Campinas, SP, and the Analytical Center and Environmental Biogeochemistry Laboratory of Embrapa Meio Ambiente in Jaguariúna, SP.

Dry mass yields were obtained gravimetrically for biochar production using pyrolysis and washing:

$$Yield (\%) = m_{BC} \times 100/m_{BM}$$
(1)

where m_{BC} and m_{BM} refers to dry masses (103 °C) before and after biochar production (pyrolysis and washing), in grams.

Recovery rates of carbon, nutrients and potentially toxic metals after pyrolysis and washing were calculated using mass balance and the contents of the chemical species of interest in the biomass and respective biochar:

Recovery (%) =
$$x_{BC}/x_{BM} \times \text{Yield}/100$$
 (2)

where Recovery corresponds to the percentage of recovery of the element after the pyrolysis process (and washing for C and nutrients), and x_{BC} and x_{BM} refers to the concentrations of each element before and after the processes for biochar production (in %, g kg⁻¹ or mg kg⁻¹, depending on the element).

Results were statistically analyzed using mean values and 95% confidence intervals.

3. Results

3.1. Physicochemical Properties

In relation to the biomasses, only BM-FC showed a pH above 7 (Table 1). PBR tend to have lower pH values, mainly due to lower quantities of inorganic cations which form basic oxides, such as Ca^{2+} , Mg^{2+} and Na^+ , as well as the presence of organic acids on their surfaces. The NPBR biomasses has a considerable amount of Ca^{2+} , Na^+ and Mg^{2+} in its compositions (shown in Table 3), resulting in biomasses with a pH higher than 7, except for BM-SS, which has a pH of 4.8 due to presence of ions Fe³⁺, which forms acid oxides in the ash content [41].

Except for FC, pH values increased with the pyrolysis process (Table 1), with the increment varying between 0.3 and 2.0. Even biomasses with alkaline pH, such as BM-PL, BM-OL, and BM-CM, the pH increased in their respective biochars. In the case of BM-FC, which initially presented the highest pH value among the biomasses, the pyrolysis process did not change it, obtaining the same value of 8.5 in BC-FC. Biochars BC-SD, BC-SB, and

BC-SS exhibited acidic pH values, although pyrolysis increased the pH relative to the respective biomasses by 2.0, 0.3 and 1.8, respectively. In BC-SB and BC-SD, the pyrolysis and washing processes increases slightly the ash and basic cations content, which elevates slightly the pH of the biochars.

Ash		Moisture		EC					
%				dS c	рп		Mass field (1)	Material	
BC	BM	BC	BM	BC	BM	BC	BM	%	
36.4 ± 1.38	21.5 ± 0.17	6.5 ± 0.18	7.9 ± 0.25	0.2 ± 0.01	2.4 ± 0.11	7.5	6.8	55.9 ± 1.52	AA
6.2 ± 0.62	2.6 ± 0.41	4.2 ± 0.19	3.9 ± 0.85	0.1 ± 0.01	0.1 ± 0.01	5.6	5.3	33.9 ± 1.39	SB
1.3 ± 0.00	0.5 ± 0.07	1.7 ± 0.05	3.8 ± 0.06	<dl< td=""><td>0.1 ± 0.01</td><td>5.3</td><td>3.3</td><td>61.2 ± 0.74</td><td>SD</td></dl<>	0.1 ± 0.01	5.3	3.3	61.2 ± 0.74	SD
54.2 ± 1.60	38.2 ± 1.91	1.2 ± 1.14	3.7 ± 0.07	0.1 ± 0.00	0.1 ± 0.01	8.5	8.5	35.4 ± 1.01	FC
44.2 ± 1.43	26.0 ± 0.93	3.7 ± 0.10	6.2 ± 0.24	0.1 ± 0.01	0.7 ± 0.01	8.6	7.7	47.2 ± 0.57	PL
22.9 ± 0.53	11.3 ± 0.43	3.9 ± 1.84	6.4 ± 0.36	0.3 ± 0.01	0.9 ± 0.05	9.1	7.6	41.2 ± 1.22	OL
51.5 ± 0.49	34.1 ± 0.69	3.3 ± 0.67	6.3 ± 0.21	0.1 ± 0.01	1.2 ± 0.09	9.3	7.4	60.6 ± 0.92	СМ
77.7 ± 0.36	62.9 ± 0.49	1.7 ± 0.15	4.2 ± 0.19	0.1 ± 0.01	0.8 ± 0.05	6.6	4.8	77.6 ± 0.01	SS

Table 1. Mass Yield, pH, EC, moisture and ashes for biomass and biochar.

<DL = Lower than the detection limit by the method.

Simultaneously, while the pH of the materials tends to increase during biochar production, the electrical conductivity of the materials decreases. The highest conductivity values were found in biomasses of NPBR, ranging from 0.7 to 1.2 dS cm⁻¹ observed in BM-PL and BM-CM, respectively. Among the PBR, only BM-AA presented high conductivity at 2.4 dS cm⁻¹, while other PBR had conductivity values about ten times lower. The highest conductivities among biochars were found in BC-AA (0.2 dS cm⁻¹) and BC-OL (0.3 dS cm⁻¹), significantly lower than those observed in biomasses. This occurs because the washing process after pyrolysis tends to remove soluble inorganic salts and smaller hydrophilic organic compounds, reducing biochar conductivity.

Regarding moisture, biochars exhibited lower levels compared to their original biomasses, while ash content tended to increase, consistent with the pyrolysis process. The retention of ash and fixed compounds in biochar directly influences the production yield, where materials with lower moisture content, lower volatile solids, and higher ash quantities result in higher biochar mass yield.

3.2. Carbon

The total C in the biomasses and biochars, with and without washing, is shown in Figure 1A, and the total C recovery in each BC, calculated using Equation 2, is shown in Figure 1B. It was observed, besides all biochars have a loss of C in the biochar production, resulting in a retaining C less than 100% (Figure 1B), the biochar production tends to maintain or increase the C content, with values of biochar between 27.87% and 81.03% (BC-FC and BC-SD, respectively) after pyrolysis (Figure 1A). This is due to biochar concentration, a phenomenon in which, despite all biomasses losing C during production, the C retained in the pyrolysis and washing processes is greater than the total mass yield in the same process, increasing the C content relative to the total mass of the biochars.

This effect was observed in five out of eight biomasses, when only FC and CM exhibits similar contents of total C recovery and mass yield, resulting in no significative changes in the total C before and after biochar production, and SS biochar exhibiting a reduction in total C content, decreasing from 17.47% to 11.90%, making it the biochar with the lowest total C content. Similar results of loss of C in biochar are observed for chicken manure [42,43] and for sewage sludge [44–46]. BM-SS and BM-CM are not constituted by lignocellulosic compounds, which resulted in biomass sources with the lowest total C

content and the highest ash content. When subjected to lower pyrolysis temperatures, they undergo degradation and depolymerization of their organic structures. However, due to their lower C content in the raw biomass, they face difficulties in recombination, leading to an accentuated volatilization process [47].





It was also observed that the washing process of biochar does not reduce the total C concentration in the biochars except for BC-FC, which had a reduction of 3.37% in total C (Figure 1A). This can be explained since bio-oil and other compounds less enriched in C are removed during washing, leaving the remaining material with a higher C content (Figure 1B). For BC-AA and BC-OL, washing resulted in a small increase in the C content in the final material, with average increases of 8.43% and 0.63%, respectively. In the other biochars, no significative changes in the total C concentration were observed as a function of washing.

Among PBR biochars, those derived from SD and SB exhibited the highest total C content, at 77.67% and 74.57%, respectively. For these two biochars, pyrolysis and washing resulted in an average total C concentration of 26.55% (averaging the %C differences between SD and SB biochars and biomass).

The biochars obtained after pyrolysis and washing from NPBR showed increases in total C content in BC-PL (from 35.27% to 42.97%) and BC-OL (from 45% to 61.37%), compared to their respective biomasses. BC-CM shown no significance in the variation of C content in biochar production (from 27.83% to 28.2%) and BC-SS showed a reduction (from 17.47% to 12.53%). However, the alteration of total C content by pyrolysis was relatively lower (from -4.93% to 16,37%) when compared to PBR-derived biochars (from -3.07% to 25.03%), especially considering those with the highest C concentration of the two categories. C recovery percentages in biochars were above 50% and reached maximum values for washed or unwashed BC-AA and for unwashed BC-FC, above 80% and 70%, respectively (Figure 1B). In general, PBR biochars exhibited higher values of C recovered after pyrolysis, compared to NPBR biochars, without the washing process.

In relation of organic C, for all PBR biochars except for FC, the retention of organic C was bigger than the mass yield (Figure 2B), leading to an concentration of organic C in the biochars. Among PBR, BC-SB exhibited 19.2% more organic C than its original biomass, while BC-AA and BC-SD showed increases of 12 and 8.8%, respectively. For BC-FC, it was observed that there was a reduction in organic C content of 7.2%, compared to its original biomass.



Figure 2. Organic C results in the biomasses and their respective biochars. (**A**) organic C content and (**B**) recovery percentage of organic C. The vertical bars represent the 5% confidence interval.

In NPBR, the remaining organic C content ranged from 41% to 60% across the four materials. Similarly to what occurred with total C in BM-FC, it was observed a reduction in organic C content in BM-CM and BM-SS. The reductions were 7.23%, 2.07%, and 5.23% for BC-FC, BC-CM, and BC-SS, respectively. The high ash content and lower C in the biomass used as feedstock to produce these biochars hinder C polymerization and recombination after the breakdown of organic molecules, promoting the volatilization of the organic fraction of the material [47].

3.3. Nitrogen

The pyrolysis and washing processes significantly altered the content and recovery of N from the biomass (Figure 3). The N contents in the PBR biomasses (Figure 3A) ranged from 0.14% to 1.69%, while in their respective biochars ranged between 0.39% and 2.48% after pyrolysis and washing. Although the biochars from PBR presented either a similar N concentration compared to their original biomass, or higher contents in relation to the



original biomass, when the recovery of N were analyzed and ranged between 38.80% and 93.46% (Figure 3B).

Figure 3. Total N results in the biomasses and their respective biochars, before and after washing. **(A)** total N content and **(B)** total N recoverey. The vertical bars represent the 5% confidence interval.

In the case of NPBR, the behavior was different. BM-CM exhibited the highest N concentration (3.49%) among the NPBR biomasses (Figure 3A), but its respective biochar had the lowest N recovery (34.54%) (Figure 3B), indicating significant losses in the pyrolysis and washing processes, which resulted in 2.29% total N in BC-CM. This biochar was the second least concentrated in N, only ahead of BC-SS, which exhibited retention similar to BC-OL and BC-PL, but since the initial N content in BM-SS was the lower in NPBR, the resulting biochar was the most depleted in N. For PL, there was no significant variation in the total N content between the biomass and the biochars after pyrolysis and washing, while N concentration was observed in BC-OL, compared to its initial biomass, due to retention of 57.07%, the highest verified among the NPBR. In general, except BC-SS, biochars produced from NPBR showed higher total N contents compared to PBR, but with much lower N retention.

For extractable N, all PBR biochars showed no significant variation or a reduction in the extractable N compared to their original biomasses (Figure 4). Sawdust and sugarcane bagasse, which naturally have low N content, exhibited N losses during biochar production (Figure 4B), but the variation in the N extractable content (Figure 4A) was maintained inside the confidence interval before and after biochar production. Among materials with higher N content, AA has the higher extractable N content and the higher remaining N among all PBR materials after biochar production. BC-FC showed a 30.7% reduction in the extractable N content, with a total of 1.04%.



Figure 4. Extractable N results in the biomasses and their respective biochars. (**A**) extractable N content and (**B**) extractable N recovery. The vertical bars represent the 5% confidence interval.

Among NPBR, extractable N contents is smaller in the biochar than the respective biomasses, and the retention were between 37% and 40% in most cases, preventing N concentration in the biochars, where the extractable N contents were 2.31%, 1.94% and 0.82% for BC-PL, BC-CM, and BC-SS, respectively. The exception was OL, which showed the higher extractable N retention during biochar production, leading to a Kjeldahl N concentration of 54.40% and the total extractable N content of 2.55%.

3.4. C/N Ratio

When analyzing the C/N ratio results, it was observed that, except for SB and SD derived materials, values ranged from 8:1 to 22:1 (Figure 5). Biochars with the highest C/N ratios were from PBR (Figure 5A), with only BC-AA showing a lower ratio than the non-plant-derived BC-OL. The OL had the highest C/N ratio among NPBR (Figure 5B) at 19:1, while biochars from other biomasses had C/N ratios between 9:1 and 12:1.

The SB exhibited a reduction in the C/N ratio from 183:1 in BM-SB to 113:1 for pre-washed and washed biochars, while SD showed a decrease from 380:1 in BM-SD to 279:1 and 228:1 after pyrolysis and washing, respectively. These materials had the lowest total N and highest total C content, reflecting these properties in the produced biochars. The biochar production process led to a greater loss of C-containing compounds than N-containing compounds in these materials, resulting in reduced C/N ratios during biochar production. In SB, the reduction of C/N ratio was 36.48%, and in SD, it was 39.81%.

In SB, N loss was lower compared to C loss during this process, reducing the C/N ratio from 183 to 113. During washing, C and N losses were similar, resulting in similar C/N ratios between washed and unwashed biochars. The BM-SD is the residue with the highest total C content and the lowest total N content resulting in a biomass with the biggest C/N ratio of 380. In the process of biochar production, it was detected the loss of C content is bigger than the N content for the saw dust, promoting the reduction of C/N ratio to 223.



Similar C and N content in saw dust and the reduction of C/N ratio in similar degrees was observed in several works [48–50].

Figure 5. C/N ratio for biomasses and the respective biochars, (A) from PBR and (B) from NPBR.

Among PBR with lower C/N ratios, AA and FC retained similar ratios throughout the biochar production process. The C/N ratio of AA was 18:1 for biomass and washed biochar. Similar results have been observed for seawater macroalgae [51] with a C/N ratio of 11:1 for raw dried *Sargassum* algae and detected an increase in the C/N ratio in biochar produced at temperatures close to 500 °C. The C/N ratio of FC varies from 19:1 (as biomass) to 22:1 (after pyrolysis and washing), with total C ranging from 30.93% to 27.87% and total N decreasing from 1.67% to 1.28%. The C/N ratio of Filter Cake and its biochar varies in the literature, from 11:1 to 35:1 [52–54]. The C content generally ranges from 28% to 35%, depending on the biochar production temperature, while N content shows significant variation, from 0.57% [54] to 3.25% [52]. This wide variation in N content is mainly influenced by the type of sugarcane processing and refining methods used.

3.5. Phosphorus

The total P quantities and quantities of this element remains in the biochar production process are influenced by the type of biomass (Table 2). It was observed that PBR biomasses and, consequently, the resulting biochars, exhibit low P content levels, where, except for BM-FC, all PBR biomasses contain less than 1.00 g kg⁻¹. In contrast, biomasses of NPBR have P content ranging from 5.46 to 26.43 g kg⁻¹, which are relevant for producing fertilizers and other products for soil application as a P source.

In only two cases the P retention was lower than close to 100% and, in both cases, the loss of P during biochar production is low. Even in cases where lower retention occurred (77.42% in BC-FC and 93.72% in BC-OL), the total P content in the biochars increased compared to the original biomasses, evidencing the pyrolysis and washing the P in the materials.

Demeinine D	Tota				
Kemaining P	Biochar	Biomass	Materiais		
%	g k				
*	2.37 ± 0.12	0.99 ± 0.02	AA		
*	0.74 ± 0.02	0.22 ± 0.02	SB		
*	0.06 ± 0.00	0.01 ± 0.00	SD		
77.42 ± 0.78	14.28 ± 0.61	11.31 ± 0.40	FC		
*	27.52 ± 1.01	13.04 ± 0.33	PL		
93.72 ± 0.62	20.75 ± 0.49	9.20 ± 0.08	OL		
*	43.46 ± 0.51	26.43 ± 0.98	СМ		
*	7.00 ± 0.13	5.46 ± 0.25	SS		

Table 2. Total phosphorus content in biomasses and respective biochars.

* no losses during biochar production. Values in parentheses represent the 5% confidence interval.

3.6. Potassium and Sulfur

Total S and K in the materials are shown in Figure 6A,C and the retention of S and K is shown in Figure 6B,D, respectively. The retention of K and S (Figure 6B,D, respectively) during the biochar production process varied depending on the biomass and biochar, however, in all cases except for SD, the biochar has less content of S and P than the respective biomass.



Figure 6. S and K results for all biochar and its respective biomasses. (**A**) Total S content, (**B**) Recovery of S, (**C**) Total K content and (**D**) Recovery of K. The vertical bars represent the 5% confidence interval.

The AA was the raw material with the highest concentrations of both K and S due to the abundance of these elements in seawater in the form of ions K⁺ and sulfates (SO_4^{2-}) [55]. However, K chlorides and sulfates, as well as most sulfates, have high melting and decomposition points while also being highly soluble in water. For this reason, these compounds remain in the product until the washing stage of biochars after pyrolysis, where they are

leached and removed, causing a drastic reduction in K and S in BC-AA, where only 23.3% of S and 3.2% of K was recovered in the biochar.

Except for AA, the PBR biomasses showed low contents of S and even lower of K. The PBR biochars had K content between 0.47 and 0.79 g kg⁻¹ and S between 0.13 and 1.00 g kg⁻¹, with the retention in S ranging from 23.18% (in BC-AA) to 39.74% (in BC-SD) after the biochar production stages, and retention in K varies considerably content ranging from 3.19% (in BC-AA) to 62.52% (in BC-SD).

Among NPBR, SS exhibited high S content mainly originating from sulfate-containing salts added during sludge conditioning (such as ferrous sulfate and aluminum sulfate), which are highly water-soluble and other S compounds which is transformed in sulfate with heating [56]. Despite remaining during the pyrolysis process, S is leached out during biochar washing, with recovery of 16.33% in the biochar production. Other NPBR biochars contain significant contents of K and S, which are present in supplements used in poultry and sheep feeding routines and are abundant in the manures from these animals when they are overfed with K and S containing supplements. The recovery of K and S during the production of BC-PL, BC-OL, and BC-CM ranged from 20.99% to 35.02% for K and from 18.76% to 31.46% for S.

3.7. Other Nutrients and Potentially Toxic Metals (PTMs)

In BC-AA, the highest contents of Ca, Mg, B, and Zn among PBR biomasses were found, along with considerable content of Mn, and only Mg salts were detected to have recovery lower than 99% out during the biochar production process, resulting in a retention of 89.8% (Table 3). In BC-SD, a significant Mn content was observed, with lower recoveries of Ca, Mn, and B, but the recovery was higher than 85% from the levels found in BM-SD. Both BC-SB and BC-FC exhibited lower recovery of the same elements, including Ca, Mg, Mn, and Zn. However, these salts leached more during the production of sugarcane bagasse biochar, primarily due to its more acidic pH, which hinders the formation of more basic salts and oxides [57]. Retention ranged from 36.7% to 56.8% in BC-SB and from 64.6% to 91.5% in BC-FC. This factor, combined with a lower amount of organic materials and higher ash content, results in a higher concentration of evaluated elements in BC-FC compared to BC-SB, making BC-FC the material with the highest levels of Fe, Mn, and Cu among all PBR biochars.

Among NPBR, higher micronutrient contents were observed compared to PBR. BC-OL exhibited the highest contents of Zn and Mn (as did all other NPBR), along with considerable contents of B and Cu. BC-OL also showed the lowest recovery of Mo (48.2% during biochar production) and higher recovery of Fe and Cu (71.2% and 89.5%, respectively). BC-PL and BC-CM, in addition to Zn and Mn contents, also contained high levels of Ca, B, and Cu and, despite lower retention of compounds when compared with BC-CM, BC-PL production resulted in retention higher than 79.4% for all evaluated elements, while BC-CM production led to a retention of only 65.7% of Cu present in BM-CM. The SS showed the highest Fe, Cu, and Zn contents among all materials; however, BC-SS production resulted in significant nutrient leaching, with retention of only 17.7% of Ca, 41.1% of Mo, 45.7% of Zn, and 67.1% of Mg.

Both biomasses and biochars showed PTM contents below the maximum limits permitted by relevant legislation [58] for the four evaluated PTMs evaluated (Table 3).

Among PBR, Cr leaching was observed during BC-SB production, accounting for approximately 67.06% of the Cr present in the biomass. For NPBR, recovery lower than 99.0% Cr was found in BC-OL and Ni in BC-PL and BC-CM, with retention of 68.0%, 76.6%, and 82.6%, respectively. Sewage sludge is the biomass with the highest contents of the four evaluated PTMs and is the only material exhibiting lower recovery of all four PTMs during

biochar production. Approximately 93.1% of Ni, 90.0% of Cd, 89.9% of Pb, and 87.9% of Cr were retained into BC-SS.

	—FC—			-SD			SB			—AA——		
R (%)	BC	BM	R (%)	BC	BM	R (%)	BC	BM	R (%)	BC	BM	PBR
						Other Nut	rients					
82.2	29.6	22	86.1	1.8	0.7	42.2	0.9	0.7	*	111.9	46.9	Ca (g kg $^{-1}$)
91.5	9.7	6.5	*	1.1	0.4	56.8	0.7	0.4	89.8	18.3	11.5	$Mg(gkg^{-1})$
*	9.3	4.1	*	0.03	0	*	0.5	0.1	*	4.9	1.2	$Fe(g kg^{-1})$
**	<12.6	<12.6	**	<12.6	24.4	**	<12.6	22.3	*	423.5	185.7	$B (mg kg^{-1})$
*	38.3	26	*	3.9	0.3	*	17.9	3.1	*	5.9	1.6	$Cu (mg kg^{-1})$
84.6	626	453.8	85.2	169.3	71.1	37.8	41.3	36	*	141	73.9	$Mn (mg kg^{-1})$
**	<1.3	<1.3	**	<1.3	<1.3	**	<1.3	<1.3	**	<1.3	<1.3	Mo (mg kg ^{-1})
64.6	19.6	18.6	*	18.6	5.3	63.3	22.2	19.9	*	30.1	9.6	$Zn (mg kg^{-1})$
						PTM						
**	< 0.4	< 0.4	**	< 0.4	< 0.4	**	< 0.4	< 0.4	**	0.6	< 0.4	$Cd (mg kg^{-1})$
*	20.5	11.4	**	0.6	< 0.5	67.1	1.5	1.5	*	6.3	2.9	$Cr (mg kg^{-1})$
**	1.7	<1.3	**	<1.3	<1.3	**	<1.3	<1.3	*	5.4	2.7	Ni (mg kg ^{-1})
**	<5	<5	**	<5	<5	**	10.6	<5	**	7.3	<5	Pb (mg kg ^{-1})
SS		CM		OL			PL					
Loss (%)	BC	BM	Loss (%)	BC	BM	Loss (%)	BC-SB	BM	Loss (%)	BC	BM	NPBK
						Other Nut	rients					
17.7	3.4	14.6	*	224.4	132.8	*	21.7	8.3	79.4	116.6	69.9	Ca (g kg $^{-1}$)
67.1	1.7	1.9	*	11.6	5.6	*	11.1	3.2	80	5.3	3.1	$Mg(gkg^{-1})$
89.9	20.7	17.8	*	2.8	0.53	71.2	1.4	0.7	*	1.1	0.1	$Fe(g kg^{-1})$
**	<12.6	<12.6	*	38.7	29.6	**	26.7	<12.6	**	39.5	<12.6	$B (mg kg^{-1})$
81.9	167.4	157.7	65.7	68.4	63.4	89.5	30.8	14.3	96.9	58.2	40.1	$Cu (mg kg^{-1})$
85.6	164	147.8	92.7	577	379.1	*	255.3	106.9	85.1	361	201.7	$Mn (mg kg^{-1})$
41.1	3.3	6.2	*	5.1	2.5	48.2	2.9	2.5	92.6	3.7	1.9	Mo (mg kg ^{-1})
45.7	1144.3	1933	94	620.7	402.1	*	430.3	177.4	91.3	470	244.9	$Zn (mg kg^{-1})$
						PTM						
90	1.4	1.2	**	< 0.4	< 0.4	**	< 0.4	< 0.4	**	< 0.4	< 0.4	$Cd (mg kg^{-1})$
87.9	85.4	75	*	8.2	4.2	68	1.8	1.1	98.4	6	2.9	$\operatorname{Cr}(\operatorname{mg} \operatorname{kg}^{-1})$
03.1	14.6	12.1	82.6	3.8	2.8	**	2.4	<1.3	76.6	2.9	1.8	Ni (mg kg ⁻¹)
95.1	11.0							110			110	

Table 3. Other nutrients and potentially toxic metals (PTM) contents and retention.

* = no losses during the biochar production, leading a retention over 99.0%. ** = one or more measurements are below the quantification limit, undermining the retention calculation.

4. Discussion

The biochar production process at low temperatures resulted in distinct products, which is expected since the final material's characteristics highly depend on the biomass used and the pyrolysis process and temperature [59]. Generally, it was found that the pyrolysis process improved, in most cases, the composition and physicochemical characteristics of the materials, which are of interest for soil use. The pH of the biochars obtained was mostly alkaline, primarily due to increased concentrations of Na, Ca, and Mg in the form of oxides, hydroxides, and carbonates [60,61]. High pH values can be advantageous for agricultural use, since a considerable amounts of soils, including Brazilian soils, are predominantly acidic [62]. Thus, their use can raise the pH, increasing the availability of nutrients and consequently promoting a healthier root environment for plants [24]. The addition of biochar, the reduction of acidity, and the consequent improvement of soil fertility have been reported by other authors [63]. Similarly to pH, biochars richer in ash can contribute to improving soil fertility [64], as they have higher nutrient contents, as observed for biochars derived from sludge, manure and some industrial residues (BC-FC, BC-SS, BC-PL, BC-CM, and BC-OL).

Another attribute of biochar with agronomic and environmental interest is its C content. Pyrolysis of lignocellulosic biomass generally produces biochars with a high fixed C content, as observed for SB and SD. For soil agricultural use, biochars with higher fixed C content tend to be more stable and less degraded, contributing to C sequestration due to the

longer retention of this chemical element in the soil without being emitted as a greenhouse gas [60]. These indicate the potential of BC-SB and BC-SD materials, particularly for soil C sequestration, as corroborated by the literature for several lignocellulosic biomasses [65].

The Intergovernmental Panel on Climate Change (IPCC) presents in its sixth assessment report, Working Group III contribution [66], biochar as a technology at maturity level TRL 6-7, ready for application in projects for carbon sequestration in the soil, with potential for global mitigation in the order of 0.3 to 6.6 Gt CO_2 year⁻¹. The high concentration of C in biochars is essential when the intuition is to account for C sequestration. In the refinement of the 2006 IPCC Guidelines for national GHG inventories [67], the default value in tier 1 for the stability of C in biochars after application to the soil for a period of 100 years is equal to 80% of the total carbon of the material. In tier 2, the fraction of stable C in biochars varies from 54 to 94%, depending on the average soil temperature and pyrolysis temperature for biochar production.

BC-SB and BC-SD also have high content of organic C, which is very important to soil structure, reducing the vulnerability of erosion and soil material loss due to weathering [68] and can promote the increment in quantity, diameter and stability of soil aggregates [69], which contributes with water retention [70], reduction of CO₂ releases [68], and improves microbial and enzymatic activities [70].

The washing process alters the C retention of the materials were, in washing of BC-SB, BC-FC and BC-SD, the washing diminished the C retention. However, the smaller recovery of carbon in washing does not alter significantly the total C content in biochars when compared with the unwashed biochar. The washing process is a important step to biochars to remove the majority of water soluble organic carbon, some of the organic acids, phenols and fulvates were are ready disponible to plants and soil microbiota, but in large quantities can diminish the stability of biochar [71]. Some of the water-soluble organic carbon can be removed in pyrolysis with inert gas flow, such as nitrogen gas or argon, but this process removes parts of organic carbon and volatile organic compounds of the biochar, diminishing the C recovery [72]. In washing process is important to remove very fine eparticles of biochar, which can be leached very easily in the water or in air during the application in the field, being a important step to biochar production for agriculture applications [71].

The highest N content after pyrolysis was observed for BC-OL among the biochars. This is consistent for biochars obtained from NPBR, since the biomasses are composed of proteins and amino acids, rather than lignocellulosic compounds. However, the remaining N fraction in biochar is stable and not readily available to plants [73].

Regarding the other macro and micronutrients, biochars are also heterogeneous. Among the PBR biochars, FC was characterized by high contents of P and other micronutrients such as iron and manganese, as indicated by the high ash content. For AA, the main characteristics observed were the metals absorbed during their time in seawater. After pyrolysis, an increase in the content of important elements such as C, P, Ca, and B were observed. The NPBRs, except for SS, presented significant contents of P, K, B, manganese, and zinc, which were reflected in the respective biochars. These materials can contribute to soil fertility and, consequently, provide nutrients for crops [74].

The production of biochar from certain biomasses can favor its use as a fertilizer or soil conditioner. For example, the application of BM-FC in soil has some disadvantages, such as excess moisture, readily decomposable organic matter, and problems with storage and application. The application of FC in soil is not economically efficient if applied more than 12 km away from the factory [75], can release odors and greenhouse gases such as N₂O and CH₄ [76], contaminate the soil with PTM and pathogens, and attract flies and other insects [75,77]. The same can be observed for BM-AA, an also a high EC [78,79].

Besides the loss of N and P (in the case of FC) and K (in case of AA), the process of biochar prouction (pyrolysis and washing) even in lower temperatures, provides stability to the material, removing excess of moisture and pathogens [80], resulting in an odorless material with a lower potential for greenhouse gas emissions [59]. Besides that, biochar production increases the shelf life of the material, resulting in a product that can be stored for a long time and transported over long distances.

The manures biomasses are rich in N, P, K, Ca and Mg, and also increase the water retention and CEC of soils, provide organic matter and have a positive effect in soil microbiome and soil respiration [81–83]. However, the use of manures as soil conditioners has similar difficulties as FC and AA.

For SS, it was observed that, although it exhibited low C, N, and pH values, its S, ash, P, and micronutrient content (zinc, copper, and manganese), along with the environmental appeal of using it in agriculture as a final disposal method, make it a material worth considering for biochar production. The BC-SS production process results in lower retention of C, N, and S, which is expected due to the volatilization of organic compounds [60,61]. However, there is an increase in pH and P content, along with a reduction in EC, indicating a viable destination for obtaining agriculturally relevant material. Thus, the production of biochar from these materials helps to improve stability and shelf life by reducing the moisture and biodegradability of the material, the release of odors, and the presence of pathogens. At the same time, it was observed in this study that pyrolysis increased the fixed C and P content, while reducing contents of S, moisture, and EC.

Regarding PTMs, the biochar production process tends to concentrate these elements since metals are not volatilized and remain in the ash portion of biochar. However, PTMs evaluated in all materials of this study were found at satisfactory quantities according to regulation No. 498 of CONAMA [58], which applies to the management of organic waste, indicating safe use in agricultural soil. Nevertheless, some elements tend to solubilize, such as Cr. For SB, Cr remains in the material after pyrolysis but is leached during BC-SB washing. The same process does not occur in FC, which, despite containing higher quantities of the same Cr species, its higher pH makes it less water-soluble and prevents it from leaching into the aqueous medium. For this reason, in large-scale BC-SB production, monitoring Cr concentration in wastewater becomes necessary.

A similar precaution applies to biochar production from NPBR, especially during washing processes, where the highest leaching of soluble salts occurs. Ni loss during biochar production from poultry residues and Cr loss in OL occurred in small amounts (0.42 mg kg⁻¹ of Ni in BC-PL production, 0.49 mg kg⁻¹ of Ni in BC-CM, and 0.36 mg kg⁻¹ of Cr in BC-OL) and are generally safe at the concentrations found in wash water. During BC-SS production, there is leaching of 8.66 mg kg⁻¹ of Cr, 3.36 mg kg⁻¹ of Pb, 0.76 mg kg⁻¹ of Ni, and 0.11 mg kg⁻¹ of Cd. When conducted on a large scale, monitoring metal concentrations in BC-LE may also be necessary.

5. Conclusions

This research showed that producing biochar via slow pyrolysis at a low temperature (300 °C) leads to notable alterations in the physicochemical properties of biomasses, with distinct outcomes based on the source of the material. Biochars from plant sources, like those created from sugarcane bagasse and sawdust, displayed elevated fixed C contents, reduced C/N ratios, and enhanced stability, positioning them as advantageous for C storage and enhancing soil structure. Conversely, NPBR biochars, such as poultry litter, sheep bedding and chicken manure, concentrated essential nutrients such as N and P while exhibiting reduced electrical conductivity and S content, making them more stable and efficient as biofertilizers.

The SS, although requiring increased monitoring due to potentially toxic metal content and leaching in wastewater during biochar production, proved relevant for agricultural applications due to its P and micronutrient contents. Slow pyrolysis at low temperatures is an efficient strategy for producing biochars with properties tailored to agricultural and environmental demands. Careful biomass selection and process control are crucial to obtaining biochars with optimized physicochemical characteristics, contributing to agricultural sustainability and solid waste management.

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Abbreviations

The following abbreviations are used in this manuscript:

- AA Arribadas Algae
- BC Biochar
- BM Biomass
- CEC Cation exchange capacity
- CM Chicken manure
- FC Filter cake
- NPBR Non-plant
- OL Sheep bedding
- PBR Plant-based residues
- PL Poultry litter
- PTM Potentially toxic metals
- SB Sugarcane bagasse
- SD Sawdust
- SS Sewage Sludge

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