



# Influence of industrial forest residue applications on *Pinus taeda*: soil, litter, growth, nutrition, and wood quality characteristics

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## Abstract

Nutrient return to soil by applying forest industry residues help ameliorate soil nutrient exhaustion and promote forest management sustainability. This study evaluated the effects of forest industry residue application on soil attributes, on forest floor amounts, and on growth and nutrition of *Pinus taeda* L. (loblolly pine). Six rates (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) of residue (boiler ash and cellulose sludge mixture) were applied to *P. taeda* stands on a low fertility soil. Seven years after application, trees were harvested for wood volume, wood biomass, canopy mass, and nutrient composition. Wood density was determined along the trunk length. Soil samples from 0–10 to 10–20 cm depths were collected for evaluating chemical attributes. Although residue application only resulted in small increases in soil Ca and P availability, high yield enhancements were observed with no compromise to wood density. Maximum growth response was obtained with the 49 T ha<sup>-1</sup> residue application. This rate enhanced trunk volume from 117 to 250 m<sup>3</sup> ha<sup>-1</sup>, total biomass from 76 to 127 T ha<sup>-1</sup>, and total C from 34.5 to 57.2 T ha<sup>-1</sup>. Residue application decreased Mn content in all biomass compartments, and values above 193.5 (needles) and 26.2 mg kg<sup>-1</sup> (bark) defined low annual growth increases. Residue application also decreased Al, Fe, S, B, Ca, and P concentrations in branches or needles. Forest floor was enhanced by residue amendment, which increased C sequestration by 7 T/ha. Forest sustainability practices can be enhanced by applications of industrial residues while improving tree yield and nutrition.

**Keywords** Southern Brazil · Loblolly pine · Biosolid waste · Soil fertility

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## Introduction

*Pinus taeda* L. (loblolly pine) is a major forest species under subtropical conditions worldwide, and the Brazilian forest industry has relied on this fast growing species (IBÁ 2019). Although *P. taeda* is well known for its high adaptability to very acidic low fertility soils (Alves et al. 2013; Motta et al. 2014), nutrient amendment to these soils can result in varied growth responses, especially in the first forest production cycle. Belote et al. (2005) suggested that management interventions at forest sites can directly affect wood quality, plantation growth rates, and wood biomass accumulation in rotations. Others have shown that nutrient amendment with different sources can result in large productivity improvement (Rodrigues 2004; Rodriguez et al. 2018), suggesting that there are conditions where lack of nutrients limit productivity (Sass et al. 2020).

Soil exhaustion has become a great concern in the forest community due to many harvest cycles without nutrient replacement (Richter et al. 1994; Motta et al. 2014; Gati-boni et al. 2020). Significant portions of nutrients remove by harvests become industrial residue wastes that are disposed in landfills and subsequently removed from natural nutrient cycling (Matysik et al. 2001; Jacobson 2003). These residue sources contain many nutrients, and application of these residues often results in larger and more frequent pine growth response compared to mineral supplement applications (Motta et al. 2014). Rodriguez et al. (2018) reported a 127% increase in pine commercial volume with the application of 84 T ha<sup>-1</sup> of pulp-mill sludge in southern Brazil. However, establishing rate recommendations can be difficult since these residues can vary widely in their nutrient composition and how they influence soil acidity (Guedes and Poggiani 2003; Rocha et al. 2004; Abreu et al. 2017). Changes in *Pinus* growth due to industrial residue application can also be associated with changes in one or more nutrients or toxic elements in tree tissues (Matysik et al. 2001; Jacobson 2003; Rodrigues 2004; Paim 2007; Sikstrom et al. 2010). However, Varnagiryte-Kabašinskiene et al. (2015) observed no changes in canopy tissue nutrient content despite growth response to ash application. Another problem is that residue can not be incorporated into soil after trees are planted; thus residue use is relegated to soil surface applications (Motta et al. 2014). In such situations, Batista et al. (2015) and Marschner and Wilczynski (1991) reported that significant portions of Ca and Mg applied as lime were found suspended in the forest floor litter and did not reach the mineral soil surface. Batista et al. (2015) also reported that the soil surface (upper few centimeters) was affected to a larger extent than subsurface soil.

Addition of nutrients to forest systems may change the quantity and quality of litterfall input and decomposition/humification rate which are major factors controlling the amount of forest floor necromass (Krishna and Mohan 2017). If nutrient amendment increases tree growth and subsequent litterfall input, it is plausible that there will be an increase in forest floor necromass (Consalter et al. 2020). In contrast, nutrient addition (or soil acidity correction) could improve litterfall composition which may enhance litter decomposition and/or promote microorganism activity thus leading to reduced forest floor necromass (Lundström et al. 2003). Such scenarios that affect forest floor necromass magnify difficulties in predicting the influence of surface applications of residues. Eleven years after cellulose residue application, Rabel et al. (2020) reported increases in forest floor necromass while others have reported decreases (Marschner and Wilczynski 1991; Lundström et al. 2003).

The goal of this study was to enhance tree growth on poor sites by applications of industrial forest residue. We hypothesized that high residue application rates would

enhance properties of very low fertility soils resulting in better tree nutrition and growth without compromising wood quality and soil protection.

## Materials and methods

### Experimental area

The experimental study site was located in *Piraí do Sul*, *Paraná* state, southern Brazil; this area was 1102 m above sea level with geographical coordinates of 24° 24' 27.36" S and 49° 58' 34.70" W. According to Köppen classification, the climate was Cfb temperate oceanic (Alvares et al. 2013; Paraná 2018a, b). Average annual precipitation during the experimental period (2011–2018) varied from 1532 to 1983 mm yr<sup>-1</sup> with an average of 90 mm for the driest months (July–August) and 190 mm for the wettest period (January) (Águas Paraná 2018a, b) (Table S1). The soil was classified as *Neossolo Quartzarênico Órtico típico* (Embrapa, 2013), equivalent to Arenosols (WRB, 2006), developed from sandstone parent material with undulated relief (Sass 2016). Prior to study initiation, two 12 year production cycles of *P. taeda* were cultivated using shallow cut management without soil amendments and fertilizers.

Before beginning the experiment, soil sampling (0–20 cm depth) was performed for chemical and granulometric characterization following methodology described by Embrapa (2009). The following results were obtained: 3.67 g dm<sup>-3</sup> of organic carbon; 4.07 of pH (CaCl<sub>2</sub>—0.01 M); 0.2 and 0.01 mg dm<sup>-3</sup> of P and Na, respectively (Mehlich-I extraction); 3.97 cmol<sub>c</sub> dm<sup>-3</sup> of (H+Al); 0.04 cmol<sub>c</sub> dm<sup>-3</sup> of Ca<sup>2+</sup>; 0.02 cmol<sub>c</sub> dm<sup>-3</sup> of Mg<sup>2+</sup>; 0.03 cmol<sub>c</sub> dm<sup>-3</sup> of K<sup>+</sup>; 0.69 cmol<sub>c</sub> dm<sup>-3</sup> of Al<sup>3+</sup>; and base saturation of 2.5%. Granulometry showed 138 g kg<sup>-1</sup> of clay, 75 g kg<sup>-1</sup> of silt, and 787 g kg<sup>-1</sup> of sand.

### Treatments and experimental design

Planting of *P. taeda* seedlings occurred in January 2011 on a spacing of 2.5 m×2.5 m. Six months after planting, industrial residue was manually applied to the soil surface (without incorporation) at the following rates: T1—control (0 T ha<sup>-1</sup>); T2—14 T ha<sup>-1</sup>; T3—25 T ha<sup>-1</sup>; T4—49 T ha<sup>-1</sup>; and T5—60 T ha<sup>-1</sup>. The experimental design was a randomized block with five replications for a total of 25 plots (plots were 156 m<sup>2</sup> in size). Each plot consisted of five rows with five trees per row. The nine central trees were considered the useful experimental area.

### Industrial residue characterization

The applied residue was a combination of cellulose sludge (70%) and boiler ash (30%), which had undergone a 12-month open-air composting process. Chemical characteristics of this industrial residue were previously described by Sass (2016) (see Table S2).

### Soil sampling and chemical analysis

In July 2018, soil was collected from all plots at depths of 0–10 cm and 10–20 cm. The following parameters were assessed: pH in 0.01 M CaCl<sub>2</sub> (1:2.5 soil: solution), Al<sup>3+</sup>, (H+Al),

Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup>, P, Zn, Mn, Fe, Cu, and C (Silva et al., 1999). Extraction of Al<sup>3+</sup>, Ca<sup>2+</sup>, and Mg<sup>2+</sup> was performed using 1 M KCl and Mehlich-I (0.025 M HCl and 0.0125 M H<sub>2</sub>SO<sub>4</sub>) was used to extract K<sup>+</sup>, P and micronutrients (Zn, Mn, Fe, and Cu). Carbon was determined after digestion with sodium dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> + H<sub>2</sub>SO<sub>4</sub>). Elements were quantified using the following techniques: Ca, Mg, Fe, Mn, Zn, and Cu—Atomic absorption spectrophotometry; C and P—Spectrophotometry/Photocolorimetry; K—Flame emission spectrometry; Al—Titration with sodium hydroxide using bromothymol blue indicator.

## Dendrometric evaluation

Seven years after planting, dendrometric evaluations were performed by examining the nine central trees in each plot. Total heights were measured with a Haglöf digital clinometer, and diameters at breast height (DBH) were evaluated using a dendrometric tape. After data collection, basal area was calculated on a 1600 trees per ha basis.

## Biomass assessment

From each experimental plot, the tree with DBH closest to the mean was selected for biomass assessment. Length and diameter of the crown was measured and separated into three parts (lower, middle, and upper third of crown). Branches were separated into live (with green needles) and dead (dry without needles). These fractions were weighed in the field, and needles from branches were separated and weighed to determine green mass. In the laboratory, samples were dried, weighed for dry mass, and processed for nutritional characterization.

Total trunk height and commercial height (total trunk minus portions less than 8 cm in diameter) were measured. Volume (with and without bark) was calculated using the methods of Hohendadl (1936). Diameters were measured at the trunk base and at points located at 10%, 30%, 50%, 70% and 90% of commercial height; using these measurements, tree volume (m<sup>3</sup>) was determined using the shape function with an adjustment value of 0.33.

Samples (5 cm thick discs) were taken at the same cut points as above, and the NBR 11,941:2003 immersion method was used for basic wood density evaluation (ABNT, 2003). Density of each segment was then extrapolated to the calculated volume (m<sup>3</sup>) of each segment.

Samples for nutrient determination in wood and bark were collected from 0% (base), 50% (middle), and 100% (commercial tip) of the trunk length. These results were used to calculate average nutrient content of trees.

## Forest floor assessment

Forest floor litter was sampled at six points in each plot using a frame (0.25 × 0.25 m). The frame was placed on the forest floor and needles along with other residue with no decomposition signs (i.e., new forest floor = new FF) were cut, manually removed, and placed into bags. The remaining materials (collectively referred to as old FF) were characterized as fragmented and partially fragmented litter (fragmented litter—F) and totally decomposed portions (humified layer—H); these samples were placed into another bag. Each old FF litter sample was placed in a container of water, manually agitated to remove soil, and floating material was manually removed into bags. Some of the suspension was gently decanted

into a fine sieve to remove any remaining soil; this process was repeated several times until all organic material was separated. All sample material was then placed into bags, dried, and weighed as done with the fresh litter. Both new FF and old FF samples were oven dried (65° C), weighed, and ground in a Wiley mill.

Total C was determined using a Perkin-Elmer CHNS/OPEZ 400 series Elemental Analyzer. In addition, color of new FF and old FF was determined according to the Munsell color system.

## Tree nutrient assessment

Needle, live branch, dead branch, bark, and wood samples were dried at 60° C, ground in a Wiley mill, and subjected to dry digestion (Martins and Reissmann 2007) before determinations of P, K, Ca, Mg, S, Fe, Mn, B and Al using an Optical Emission Spectrometer with Inductively Coupled Plasma (ICP–OES Varian, 720–ES). Carbon and N analyses were done using a Perkin-Elmer CHNS/OPEZ 400 series Elemental Analyzer.

Masses (dead branches, live branches, needles, bark and wood) and corresponding average nutrient concentrations were used to calculate nutrient content in each tree part. Values were extrapolated to a hectare basis based on a population of 1,600 trees per ha.

## Statistical analysis

Soil characteristics, litter, nutrient concentration, and nutrient content data were subjected to the Bartlett test followed by analysis of variance (ANOVA), and Tukey's test using a 95% confidence level ( $p < 0.05$ ). Regression analyses were done using DBH and height or volume, and when significant, the Maximum Technical Efficiency rate (MTE) was calculated using the first derivative of the second-degree equation ( $X = -b1 / 2.b2$ ). The annual average increment (AAI) was calculated by dividing final tree volume by the age of the *P. taeda* plantation. Regression trees were utilized to predict the annual average increment (AAI). Statistical analyses were performed using R software (version 3.5.3) and regression tree was performed by using the function *ctree* of “party” package (Hothorn et al., 2006).

## Results

### Soil proprieties

No major changes in soil chemical proprieties were observed seven years after residue application (Table 1). Soil pH varied by depth, where the 0–10 cm layer was slightly higher (average value of 4.08) compared to the 10–20 cm layer (average value of 3.90). The same was observed for exchangeable Al. At the 10–20 cm depth, the H+ Al and Ca concentration had the highest value at the 49 T ha<sup>-1</sup> residue rate. This was also true for P concentration at the same soil depth.

Phosphorus increased from 2.64 mg kg<sup>-1</sup> (control) to 14.24 mg dm<sup>-3</sup> (49 T ha<sup>-1</sup>) at the 10–20 cm soil depth, and a small increase from 1.72 mg dm<sup>-3</sup> (control) to 4.42 mg dm<sup>-3</sup> (49 T ha<sup>-1</sup>) was seen in the upper layer (0–10 cm). Residue application did not change soil C concentration, but average values at the 0–10 cm layer (18.9 g dm<sup>-3</sup>) were lower compared to those observed in the 10–20 cm layer (24.8 g dm<sup>-3</sup>) (Table 1).

**Table 1** Chemical characteristics of soil (0–10 and 10–20 cm depths) in a 7 year-old *Pinus taeda* stand cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) on a low fertility soil in Piratí do Sul, Paraná state, southern Brazil

	Rate (T ha <sup>-1</sup> )	pH CaCl <sub>2</sub>	Al cmol <sub>c</sub> (cmol <sub>c</sub> dm <sup>-3</sup> )	H+Al	Ca	Mg	K	Na	P (mg dm <sup>-3</sup> )	C (g dm <sup>-3</sup> )
0–10 cm	0	4.01 a A	0.78 a B	5.00 ab B	0.14 a A	0.10 a B	0.04 a B	0.02 a A	1.72 b B	25.12 a B
	14	4.08 a A	0.62 a B	4.08 b B	0.24 a A	0.12 a B	0.04 a B	0.02 a A	2.18 b B	14.42 a B
	25	4.11 a A	0.63 a B	4.24 b B	0.20 a A	0.08 a B	0.04 a B	0.02 a A	3.28 ab B	21.10 a B
	49	4.02 a A	0.77 a B	6.18 a B	0.28 a B	0.12 a B	0.04 a B	0.02 a A	4.42 a B	22.18 a B
	60	4.20 a A	0.66 a B	5.16 ab B	0.24 a A	0.12 a B	0.04 a B	0.02 a A	3.26 ab B	11.80 a B
10–20 cm	0	3.82 a B	1.32 a A	7.36 ab A	0.14 b A	0.14 a A	0.04 a A	0.02 a A	2.64 b A	22.78 a A
	14	3.95 a B	0.91 a A	4.92 b A	0.28 b A	0.12 a A	0.05 a A	0.03 a A	4.18 b A	21.48 a A
	25	3.96 a B	1.07 a A	5.84 b A	0.38 ab A	0.14 a A	0.05 a A	0.03 a A	6.44 ab A	27.00 a A
	49	3.92 a B	1.20 a A	8.58 a A	0.76 a A	0.16 a A	0.08 a A	0.02 a A	14.24 a A	31.50 a A
	60	3.85 a B	0.95 a A	6.18 ab A	0.34 ab A	0.14 a A	0.05 a A	0.02 a A	5.22 ab A	21.48 a A
	CV (a) %	4.42	27.1	28.3	93.8	33	31.8	37.7	87.1	47.5
	CV (b) %	3.02	16.9	33.9	59.3	41.1	24	27.7	75.1	44.4

Means followed by the same letters in a column do not differ statistically at the 5% probability level (Tukey's test)

Lower case letters refer to statistical differences between treatments. Capital letters refer to statistical differences between depths

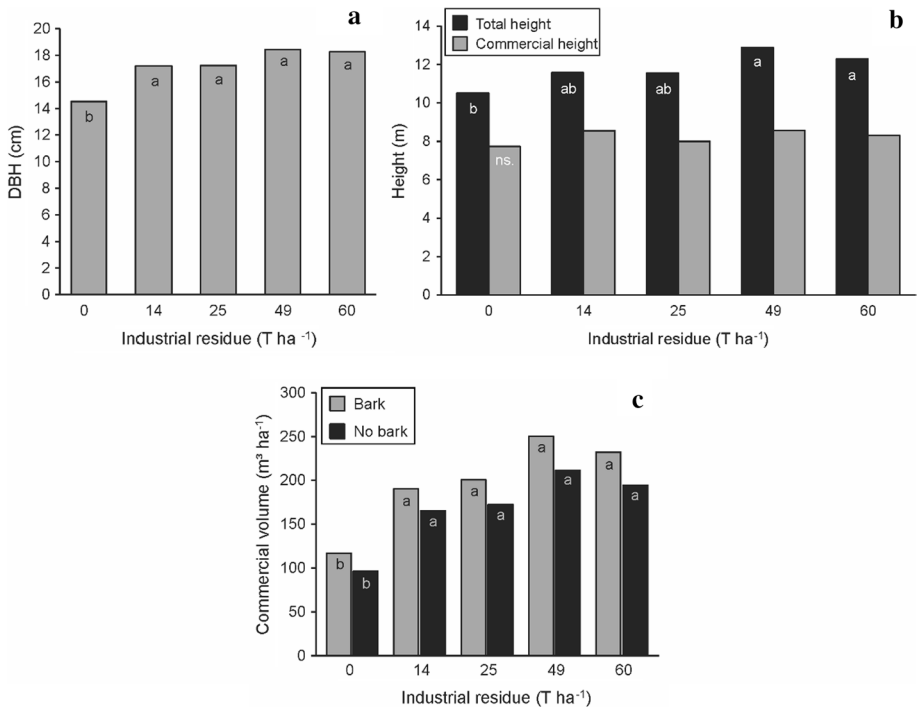
CV (a)% treatment variation coefficient, CV (b)% depth variation coefficient

## Tree growth

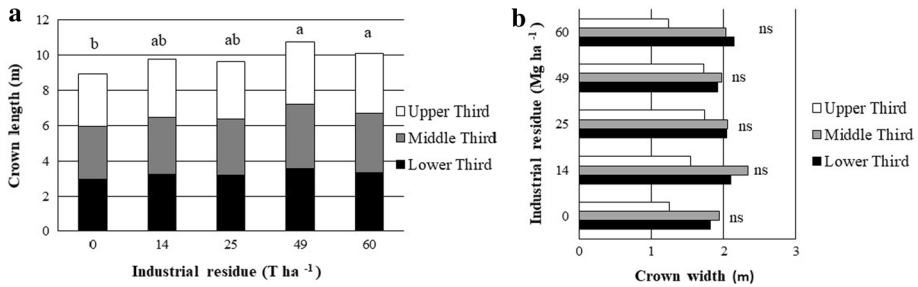
Diameter at breast height was positively influenced by residue treatments ( $p < 0.01$ ) with no difference among rates (Fig. 1a). The highest value was observed at the 49 T ha<sup>-1</sup> rate (attaining a diameter of 18.44 cm), which represented a gain of ~4 cm relative to the control (Fig. 1a). However, average commercial tree height was not affected by residue application ( $p = 0.22$ ; Fig. 1b) despite some increases in total tree height at higher rates (Fig. 1b). Similarly, trunk volume was influenced by residue without difference among rates. Volume without bark ( $p < 0.01$ ) increased from 96 to 211 m<sup>3</sup> ha<sup>-1</sup> and volume with bark ( $p < 0.01$ ) increased from 117 to 250 m<sup>3</sup> ha<sup>-1</sup> (Fig. 1c), with the highest value observed at 49 T ha<sup>-1</sup>. Thus, the Annual Average Increment (AAI) for volume without bark was 13.7 m<sup>3</sup> ha<sup>-1</sup> year<sup>-1</sup> in the control treatment and 30.2 m<sup>3</sup> ha<sup>-1</sup> year<sup>-1</sup> for the 49 T Mg ha<sup>-1</sup> rate (i.e., AAI increased more than two times).

Tree crown length was influenced by residue use ( $p = 0.01$ ) and reached an average of 10.4 m at the two highest rates, compared to 8.9 m for the control (Fig. 2a). For crown width, residue application did not result in differences; averages were 2.00 m for the lower ( $p = 0.49$ ) and middle ( $p = 0.69$ ) thirds, and 1.50 m for the upper third ( $p = 0.80$ ) (Fig. 2b).

Total tree biomass increased from 75.71 (control) to 127.39 T ha<sup>-1</sup> (49 T ha<sup>-1</sup> rate), and wood biomass (trunk without bark) increased from 48.91 (control) to 84.12 and 77.30 T ha<sup>-1</sup> (Table 2), for 49 and 60 T ha, respectively. There was no difference among



**Fig. 1** Diameter at breast height—DBH (a), total and commercial height (b) commercial volume with bark and commercial volume without bark (c) of 7-year-old *Pinus taeda* at different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>)



**Fig. 2** Crown length (a) and crown width (b) of 7 year-old *Pinus taeda* at different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>). Column means with the same letters do not differ statistically at the 5% probability level (Tukey's test). ns = not significant

tree biomass components (wood, bark, needles and live and dead branches) as a result of industrial residue amendment (Table 2). Regardless of treatment, the decreasing sequence for total tree biomass components was: wood (64.60–68.99%) > bark (10.28–14.40%) > live branches (8.85–11.65%) > needles (4.55–7.98) > dead branches (3.20–7.85%) (Table 2).

### Wood quality

Wood density varied with trunk height ( $p < 0.01$ ), decreasing from the base to the top of the tree (Table 3). This also decreased for the highest treatment ( $p = 0.02$ ), but there was no interaction with height vs residue application ( $p = 0.83$ ). However, weighted density (due to differences in volume of trunk segments) did not show significant variation with residue application, and density values were close to ideal for the pulp and paper industry (0.405 g cm<sup>-3</sup>).

### Tree tissue elemental composition

There was no effect of residue application on concentrations of C, N, K, Mg and S in tree tissues (Table 4). Phosphorus, Ca, and S concentrations showed the highest values in needles of the control; this was also true for P, Mg, and S in live branches. Concentrations of Fe, Mn, B, and Al decreased with increasing residue rate (Table 4). For bark and wood, only Mn concentration was decreased by residue application (Table 5). Manganese was the only element that displayed decreases in all five evaluated tissues types with residue application (Tables 4 and 5). Regression trees confirm the importance of Mn nutrition in tree growth; among all elements analyzed, only Mn was a predictor of AAI. Manganese concentrations below 193.5 mg kg<sup>-1</sup> in needles were associated with the highest AAI (Fig. 3a); similarly, bark concentrations lower than 26.17 mg kg<sup>-1</sup> were also associated with the highest AAI (Fig. 3b).

### Forest biomass nutrient content

The structural elements C and N increased in bark and wood fractions as productivity increased in response to residue application (Table S4). Potassium, Ca, Mg, and S content increased only in the wood fraction (Table S4). In contrast, micronutrient and Al decreases



**Table 2** Compartmentalization of biomass in 7 year-old *Pinus taeda* trees cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) in Piraf do Sul, Paraná state, southern Brazil

Rate (T ha <sup>-1</sup> )	Lower third (T ha <sup>-1</sup> )	Middle third	Upper third	Total	Relative biomass (%)
<i>Needles</i>					
0	0.54	3.29	2.21	6.04	7.98
14	0.31	2.38	4.13	6.82	6.61
25	0.12	3.19	2.12	5.43	5.36
49	0.16	3.37	5.20	8.73	6.85
60	0.11	1.74	3.25	5.10	4.55
<i>Live branch</i>					
0	2.26	4.27	1.57	8.1	10.7
14	2.22	6.11	3.69	12.02	11.65
25	0.97	6.20	2.18	9.35	9.23
49	1.32	8.03	4.48	13.83	10.86
60	1.75	4.87	3.30	9.92	8.85
<i>Dead branch</i>					
0	3.83 ab	0.04	0.00	3.84	5.07
14	8.12 a	0.00	0.00	8.10	7.85
25	5.48 ab	0.00	0.00	5.50	5.43
49	5.10 ab	0.96	0.00	6.06	4.76
60	3.40 b	0.19	0.00	3.59	3.20
<i>Tree canopy biomass (needles + live and dead branch)</i>					
0	6.6	7.6	3.78	17.98 b	23.75
14	10.63	8.49	7.82	26.94 ab	26.11
25	6.59	9.39	4.3	20.28 ab	20.02
49	6.58	12.36	9.68	28.62 a	22.47
60	5.26	6.8	6.55	18.61 ab	16.61
<i>Wood (barkless trunk)</i>					
0	12.96 b	25.91	10.04 b	48.91 b	64.60
14	22.08 ab	29.15	14.38 ab	65.61 ab	63.60
25	23.42 ab	29.81	15.96 a	69.19 ab	68.30
49	29.20 a	37.12	17.80 a	84.12 a	66.03
60	26.40 a	34.11	16.79 a	77.30 a	68.99
<i>Bark</i>					
0	3.51 b	4.23	1.08 b	8.82 b	11.65
14	5.62 ab	3.98	1.01 b	10.61 ab	10.28
25	5.58 ab	4.82	1.44 ab	11.84 ab	11.69
49	8.01 a	5.21	1.43 ab	14.65 ab	11.50
60	7.63 a	6.71	1.79 a	16.13 a	14.40
<i>Trunk biomass (barkless trunk and bark)</i>					
0	16.47 b	30.14	11.12 b	57.73 b	76.25
14	27.7 ab	33.13	15.39 ab	76.22 ab	73.89
25	29.00 a	34.63	17.40 a	81.03 ab	79.98
49	37.21 a	42.33	19.23 a	98.77 a	77.53
60	34.03 a	40.82	18.58 a	93.43 a	83.39

**Table 2** (continued)

Rate (T ha <sup>-1</sup> )	Lower third (T ha <sup>-1</sup> )	Middle third	Upper third	Total	Relative biomass (%)
<i>Tree biomass (needles + live and dead branch + barkless trunk and bark)</i>					
0	23.07 b	37.74	14.90 b	75.71 b	100
14	38.33 a	41.62	23.21 ab	103.16 ab	100
25	35.59 ab	44.02	21.70 ab	101.31 ab	100
49	43.79 a	54.69	28.91 a	127.39 a	100
60	39.29 a	47.62	25.13 a	112.04 a	100

**Table 3** Wood density at different heights, basic average per plant, and weighted density of 7 year-old *Pinus taeda* trees cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) applied to a low fertility soil at Piraí do Sul, Paraná state, southern Brazil

Relative height (%)	Rate (T ha <sup>-1</sup> )					Average
	0	14	25	49	60	
	g cm <sup>-3</sup>					
10	0.472 <sup>ns</sup>	0.444	0.450	0.444	0.428	0.448a
30	0.434 <sup>ns</sup>	0.388	0.404	0.404	0.400	0.406b
50	0.410 <sup>ns</sup>	0.364	0.372	0.372	0.384	0.380bc
70	0.392 <sup>ns</sup>	0.366	0.382	0.364	0.348	0.370c
90	0.374 <sup>ns</sup>	0.392	0.356	0.368	0.376	0.373c
Basic average	0.416a	0.390ab	0.393ab	0.390ab	0.387b	
Weighted density	0.429 <sup>ns</sup>	0.399	0.416	0.403	0.398	

were observed only in crown compartments (i.e., needles and live and dead branches). In these three fractions, Mn and B content decreased with residue application, Al decreased in dead branches and needles, and Fe decreased in dead branches (Table S3).

Macronutrients, micronutrients, and Al content in aboveground tree biomass followed the sequence C > N > K > Ca > P > Mg > S for macronutrients, and Al > Mn > Fe > B for micronutrients and Al (Table S4). Macronutrients and Al amounts mirrored biomass production, and attained highest values with the rate (49 T ha<sup>-1</sup>) that produced maximum growth. There was a decrease in total Mn and B in aboveground parts, suggesting that large decreases in tissue concentration accompanied increased growth.

## Forest floor

The residue treatment that resulted in maximum growth increased the amount of necromass in both old FF and total litter (Table 6). The relative increase was larger for old FF (23.8 to 46.0 T ha<sup>-1</sup>) than for new FF (5.1 to 6.3 T ha<sup>-1</sup>). In general, increased necromass was not associated with amendment rate.

Regardless of treatment, all new FF was Reddish brown after grinding, indicating no influence of residue application. However, ground old litter was observed to have changed color with residue application. The control had color between Dark reddish gray and Dark

**Table 4** Nutrient concentration in branches (dead and live) and needles of 7 year-old *Pinus taeda* trees cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 Mg ha<sup>-1</sup>) applied to a low fertility soil at Pirai do Sul, Paraná state, southern Brazil

Rate (T ha <sup>-1</sup> )	N (g kg <sup>-1</sup> )	C	P	K	Ca	Mg	S	Fe (mg kg <sup>-1</sup> )	Mn	B	Al
<i>Dead branches</i>											
0	2.25 a	471 a	0.13 a	0.63 a	1.46 a	0.23 a	0.11 a	36.74 a	296.00 a	89.01 a	228.75 a
14	2.40 a	459 a	0.07 a	0.31 a	1.37 a	0.16 a	0.04 a	24.92 ab	94.25 b	84.51 a	151.91 b
25	2.30 a	475 a	0.08 a	0.53 a	1.40 a	0.21 a	0.03 a	35.42 ab	97.55 b	42.89 ab	170.51 ab
49	1.99 a	473 a	0.07 a	0.87 a	1.38 a	0.20 a	0.02 a	13.65 b	51.64 b	26.70 b	106.00 b
60	3.02 a	454 a	0.07 a	0.80 a	1.34 a	0.21 a	0.03 a	14.49 b	41.57 b	20.33 b	107.41 b
Average	2.392	466	0.084	0.628	1.39	0.202	0.046	25.04	116.20	52.69	152.92
<i>p</i> -value	0.13	0.08	0.27	0.468	0.945	0.111	0.199	0.01	<0.01	<0.01	<0.01
CV%	94.68	3.05	57.45	82.39	15.44	21.47	139.17	56.21	87.16	72.04	37.1
<i>Live branches</i>											
0	5.00 a	465 a	0.39 a	1.74 a	1.26 a	0.30 a	0.08 a	27.90 a	202.25 a	86.97 a	154.10 a
14	5.41 a	466 a	0.24 b	1.59 a	1.34 a	0.23 b	0.06 ab	23.62 a	97.22 b	72.38 a	137.43 ab
25	4.95 a	472 a	0.24 b	1.64 a	1.29 a	0.26 ab	0.05 bc	22.46 ab	88.59 b	40.97 b	122.21 abc
49	4.93 a	469 a	0.17 b	1.65 a	1.30 a	0.26 ab	0.04 bc	15.11 bc	51.18 c	27.45 bc	96.85 c
60	4.06 a	441 b	0.17 b	1.55 a	1.30 a	0.25 ab	0.04 c	14.89 c	48.03 c	18.68 c	105.68 bc
Average	4.87	462.60	0.24	1.63	1.30	0.26	0.05	20.80	97.45	49.29	123.25
<i>p</i> -value	0.84	<0.01	<0.01	0.52	0.96	0.03	<0.01	<0.01	<0.01	<0.01	<0.01
CV%	65.98	32.1	36.04	19.73	24.25	21.3	28.44	33.6	32.85	29.94	25.33
<i>Needles</i>											
0	12.59 a	463 a	1.10 a	3.57 a	1.89 a	0.57 a	0.16 a	40.98 a	613.41 a	93.28 a	405.39 a
14	10.40 a	465 a	0.79 b	3.60 a	1.43 b	0.45 a	0.12 ab	24.92 b	222.82 b	73.57 b	250.37 b
25	11.07 a	468 a	0.73 b	3.64 a	1.32 b	0.49 a	0.11 b	28.54 b	206.85 b	45.06 a	230.31 bc
49	10.82 a	440 a	0.57 b	3.75 a	1.41 b	0.46 a	0.09 b	21.67 b	147.58 b	32.84 cd	184.62 c
60	12.64 a	436 a	0.69 b	3.94 a	1.38 b	0.51 a	0.10 b	24.97 b	144.18 b	21.98 d	213.65 bc
Average	11.5	455	0.78	3.70	1.49	0.50	0.12	28.22	266.97	70.64	256.87

**Table 4** (continued)

Rate (T ha <sup>-1</sup> )	N (g kg <sup>-1</sup> )	C	P	K	Ca	Mg	S	Fe (mg kg <sup>-1</sup> )	Mn	B	Al
<i>p</i> -value	0.3	0.3	<0.01	0.22	<0.01	0.04	<0.01	<0.01	<0.01	<0.01	<0.01
CV%	30.4	10.96	31.21	13.97	28.95	22.75	36.06	32.21	25.21	22.95	21.21

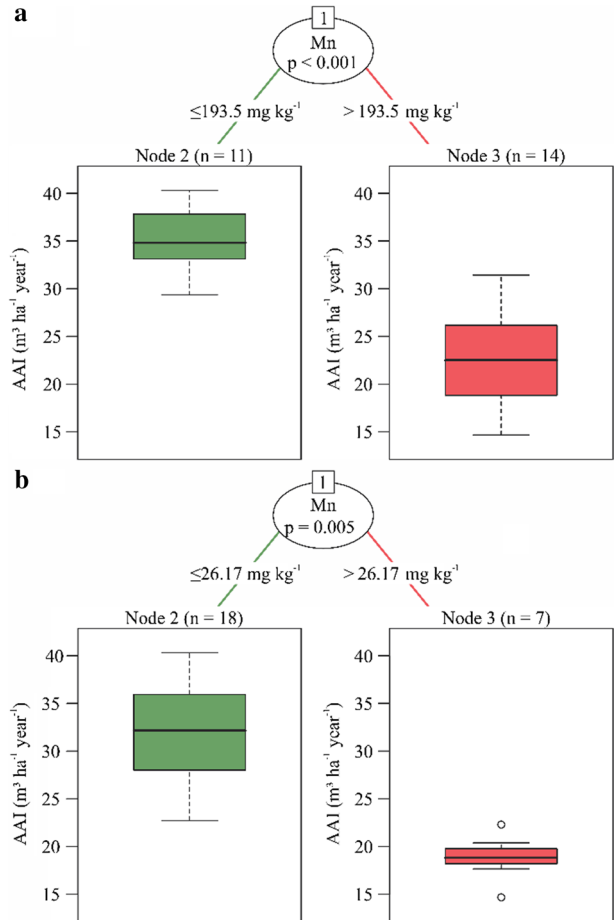
Means followed by the same letters in a column do not differ statistically at the 5% probability level (Tukey's test)

**Table 5** Nutrient concentration in bark and wood of 7 year-old *Pinus taeda* trees cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) applied to a low fertility soil at Pirai do Sul, Paraná state, southern Brazil

Rate (Mg ha <sup>-1</sup> )	N (g kg <sup>-1</sup> )	C	P	K	Ca	Mg	S	Fe (mg kg <sup>-1</sup> )	Mn	B	Al
<i>Bark</i>											
0	2.56 a	426.87 a	0.19 a	0.76 a	0.42 a	0.15 a	0.28 a	67.01 a	60.62 a	11.72 a	479.84 a
14	2.91 a	428.33 a	0.25 a	0.74 a	0.52 a	0.16 a	0.12 a	55.72 a	43.68 b	11.71 a	476.95 a
25	2.77 a	415.93 a	0.19 a	0.64 a	0.47 a	0.15 a	0.21 a	64.59 a	36.53 bc	13.25 a	473.28 a
49	2.77 a	432.34 a	0.21 a	0.88 a	0.69 a	0.19 a	0.22 a	63.42 a	33.70 bc	11.89 a	447.68 a
60	2.62 a	326.27 a	0.18 a	0.80 a	0.66 a	0.15 a	0.28 a	61.79 a	26.58 c	11.44 a	444.18 a
Average	2.72	405.94	0.2	0.76	0.55	0.16	0.22	62.5	40.22	12	464.38
p-value	0.28	0.93	0.11	0.56	0.03	0.31	0.76	0.95	<0.01	0.58	0.83
CV%	17.03	5.53	36.34	50.89	47.53	36.1	161.62	63.73	27.69	27.3	23.43
<i>Wood</i>											
0	1.83 a	457.37 a	0.19 a	1.07 a	0.46 a	0.17 a	0.04 a	3.38 a	113.51 a	4.15 a	38.07 a
14	1.89 a	450.32 a	0.19 a	1.05 a	0.50 a	0.17 a	0.05 a	5.75 a	90.47 b	3.96 a	40.04 a
25	1.69 a	453.89 a	0.18 a	1.00 a	0.49 a	0.16 a	0.04 a	4.22 a	85.75 b	3.56 a	34.89 a
49	1.71 a	454.13 a	0.18 a	1.12 a	0.54 a	0.18 a	0.05 a	3.12 a	60.61 c	4.13 a	42.41 a
60	1.88 a	454.46 a	0.19 a	1.14 a	0.56 a	0.17 a	0.04 a	3.70 a	54.89 c	3.91 a	39.58 a
Average	1.8	454.03	0.18	1.07	0.51	0.17	0.04	4.03	81.05	3.94	38.99
p-value	0.14	4386	0.75	0.24	0.06	0.1	0.13	0.07	<0.01	0.89	0.73
CV%	14.59	1.78	17.93	16.42	20.7	13.94	20.83	65.9	23.04	43.64	38.84

Means followed by the same letters in a column do not differ statistically at the 5% probability level (Tukey's test)

**Fig. 3** Regression tree of annual average increment (AAI) explained by elemental composition of needles (a) and base bark (b)



gray, while treatments receiving residue varied between Dark gray and Black (Table S5); color of old FF also varied among individual samples (Table S5). The authors believe that small charcoal fragments found in applied residue may have influenced color changes.

Concentrations of C in new FF (437–460  $\text{g kg}^{-1}$ ) were higher than those observed in old FF (318 to 350  $\text{g kg}^{-1}$ ). There was no effect of residue amendment on C concentration for new FF and old FF. Consequently, total carbon content in litter increased with residue application, with an increase of ~60% at 49  $\text{T ha}^{-1}$  compared to the control.

## Discussion

### Soil proprieties

Despite amount of residue applied, enhancement was only observed for Ca and P at 10–20 cm depth. Although Ca availability was more than doubled, values were still considered very low ( $< 0.5 \text{ cmol}_c \text{ dm}^{-3}$ ) or low ( $0.5\text{--}1.0 \text{ cmol}_c \text{ dm}^{-3}$ ) (Pauletti and Motta 2018), while P availability increased from very low ( $< 2.0 \text{ mg dm}^{-3}$ ) to low ( $2.0\text{--}3.0 \text{ mg dm}^{-3}$ )

**Table 6** Carbon concentration and content, dry mass, and color of new FF, old FF, and total FF litter from a *P. taeda* forest cultivated under different rates of industrial residue (0, 14, 25, 49, and 60 T ha<sup>-1</sup>) applied to a low fertility soil at Piraf do Sul, Paraná state, southern Brazil

Rate T ha <sup>-1</sup>	C g kg <sup>-1</sup>	Dry mass T ha <sup>-1</sup>	C kg ha <sup>-1</sup>	Color
<i>New FF<sup>1</sup></i>				
0	460 n.s	5.1 n.s	2,357 n.s	Reddish brown
14	457	6.3	2,879	Reddish brown
25	437	6.3	2,756	Reddish brown
49	451	6.0	2,690	Reddish brown
60	455	6.1	2,746	Reddish brown
<i>Old FF</i>				
0	350 n.s	23.8 b	8,317 b	Reddish brown /Dark reddish gray/Dark gray
14	318	39.5 ab	12,835 ab	Dark gray/Dark reddish brown
25	331	30.2 ab	10,038 ab	Dark gray/Very dark gray/Dark reddish gray
49	332	46.0 a	15,231 a	Dark gray/Very dark gray/Black
60	349	39.8 ab	13,988 ab	Dark gray/Very dark gray/Dark reddish brown
<i>Total FF</i>				
0	370 n.s	28.9 b	10,674 b	
14	340	45.8 ab	15,714 ab	–
25	351	36.5 ab	12,794 ab	–
49	347	52.0 a	17,921 a	–
60	364	45.8 ab	16,734 ab	–

<sup>1</sup>FF=forest floor—organic layer of residue, combination of fresh and fragmented and humidified plant material

and medium (4.0–5 mg dm<sup>-3</sup>). Thus, observed improvements are probably attributable to P availability, which has been shown to be the element most limiting *P. taeda* growth (Con-salter et al. 2020; Sikstrom et al. 2010).

In general, when there is no incorporation of residue and lime into soil, effects are concentrated in the first few centimeters of the upper soil profile. Eleven years after broadcasting 30 T ha<sup>-1</sup> of lime over the forest floor, large acidity variations found in forest floor litter and in the 5 cm soil depth prompted Meiwes (1995) to state that decades may be needed to change acidity in deeper soil layers. Conversely, Marschner and Wilczynski (1991) applied 6.1 T ha<sup>-1</sup> of lime to a 40 year-old forest and found soil changes to a depth of 10 cm after 3 years. These varied findings highlight the necessity for more studies examining the influence of residue on deep soil layers.

Batista et al. (2015) and Rabel et al. (2020) found that thick forest floor litter can be a barrier to industrial residue and lime applications. However, residue application in our study occurred in the absence of significant litter amounts to compromise soil contact and reaction with soil. In addition, the low adsorption capacity and small buffer capacity due to low clay concentration (13%) were excellent soil characteristics for observing changes in soil properties. Thus, the lack of large changes in soil properties demonstrates the low potential of our residue for correcting soil acidity and supplying nutrients.

A previous evaluation of soil fertility in this study area found no change in pH, Ca, and available P for three consecutive years following residue application (Sass et al. 2020).

Thus, changes observed in soil 7 years after application indicated a slow release of nutrients from applied residue. Rabel et al. (2020) evaluated the effects of using alkaline residues (from recycled paper) after 1, 5, and 10 years and found that the greatest change occurred 5 years after application, which also suggested slow reaction. Unlike lime sludge (Corrêa et al. 2007), residue used in our study had no acidity correction capacity, and unlike sewage sludge (Ibrahim et al., 2019), our residue was nutrient poor.

## Tree growth

In contrast to minimal changes observed in soil, tree growth response was notable; volume increased more than 100% in the 49 T ha<sup>-1</sup> treatment. It is important to highlight that residue application only increased Ca and P; concentrations of both nutrients were more than doubled. This beneficial effect was not unexpected since this area suffered from a combination of naturally low soil fertility and nutrient exhaustion from successive *Pinus* cultivation without fertilization or liming (Gatiboni et al. 2020). These responses to residue confirm the high growth potential and the lack of limiting factors (water availability and rooting soil depth) in this area.

Large yield enhancements have been observed under similar soil conditions and management (Rodrigues 2004; Rodriguez et al., 2018). Rodrigues (2004) also observed gains of 80% in height, DBH, and volume in response to application of industrial residue (20 T ha<sup>-1</sup>) on low fertility sandy soils. Likewise, working with *P. taeda* on a sandy soil, Rodriguez et al. (2018) found that higher rates of composted cellulose sludge (84 T ha<sup>-1</sup>) increased tree diameter, height, and volume by 24, 37, and 127%, respectively. Sikstrom et al. (2010) found a sixfold volume improvement in *Pinus sylvestris* for a drained peat area amended with ash. Others reported increases close to 15% with residue application on clay-loam (Rabel et al. 2020) and clayey soils (Paim 2007). Results of Rodriguez et al. (2018) confirm the potential of applying cellulose sludge as a fertilizer to improve wood productivity of forest plantations established on low fertility soils. In general, growth improvements have been observed in *P. taeda* plantations under conditions without climatic and depth limitations in nutrient poor soils receiving fertilizer (Samuelson et al. 2004; Albaugh et al. 2020).

## Biomass partitioning

Unlike our study where biomass abundance followed the sequence wood > branches (live and dead) > bark > needles, Corrêa and Bellote (2011) found wood > bark > branches > needles for sites with 10.5 to 12 year-old *Pinus caribaea* var. *hondurensis*. Albaugh et al. (2004) also reported differences for *P. taeda* where 8 year-old trees followed the sequence stem > branch > needles > bark and 16 year-old trees had a sequence of wood > branch > bark > needles. They also reported a sharp decrease for needle (20–5%) and increase for stem (49–58%) contributions to total aboveground biomass in these respective age classes. Thus, our 7 year-old *P. taeda* system displayed similarities to 16 year-old trees evaluated by Albaugh et al. (2004).

We noted no differences in crown tree partitioning as a result of residue amendment. This is contrary to the general statement that fertilization or better fertility conditions increases stem partitioning to tree crown biomass. Corrêa and Bellote (2011) found 57, 22, 12, and 8% for poor growth sites compared to 67, 18, 8, and 8% for good growth sites for stem, bark, branches, and needles of *Pinus caribaea* var. *hondurensis*, respectively.



Albaugh et al. (2004) reported changes in stem partitioning from 58 to 63% in tree crown as result of fertilization in a 16 year-old *P. taeda* forest. However, Samuelson et al. (2004) found no differences in tree crowns (foliage, live branches and stem) of six year-old *P. taeda*. However, our higher study rate promoted a decrease in tree growth (likely due to some nutritional imbalance) similar to that observed by Rodrigues (2004).

## Wood quality

Although increased growth of *P. taeda* in response to residue application resulted in decreased average wood density, weighted density was unaffected. This variation may be associated with tree growth rate, where higher growth rate results in lower density (Pretzsch et al. 2018; Antony et al. 2009). However, Castelo et al. (2008) observed the opposite for 14, 16, and 18 year-old *P. taeda* (i.e., higher growth rate resulted in higher density), while Albaugh et al. (2004) found no variation in wood density due to fertilization. It is important to note that our 7 year-old trees had not reached normal harvest age (typically 15 years in Brazil) and that older trees have more adult wood which tends to have higher density (Latorraca and Albuquerque 2000). Since wood density remained very close to and sometimes surpassed values of the pulp industry ( $0.405 \text{ g cm}^{-3}$ ), the double increase in growth did not decrease tree quality.

## Tissue elemental composition

Residue application caused Mn to vary more than other elements in plant tissues; this element decreased in needles, dead branches, live branches, bark, and wood. Rodriguez et al. (2018) found decreased Mn concentration in *P. taeda* woody tissue. Toxic effects of Mn have been observed in a gymnosperm native to southern Brazil (*Araucaria angustifolia* Bert. O. Ktze.), with an inverse relationship being observed between needle Mn concentration and growth (Hoogh 1981). This was also true in the present study based on regression tree results. Motta et al. (2014) indicated that low growth sites on acidic soils were characterized by high Mn and low Fe in leaf tissue. Given high soil acidity, Gomes et al. (2019) suggested that *Pinus* species are more sensitive to Mn than Al indicating possible Mn toxicity or imbalance. Analyzing *Pinus oocarpa* and *Pinus caribaea var hondurensis*, Gomes et al. (2019) found a fivefold increase in needle Mn concentration when soil pH was reduced from 5 to 4. Sikstrom et al. (2010) reported increased Mn in *Pinus sylvestris* leaf tissue due to use of alkaline ash. Working with *P. taeda* seedlings, South et al. (2017) did not observe changes in leaf Mn concentration with pH variation due to addition of acidifiers and acidity correctors. In the current study, despite no change in soil pH, residue addition was associated with less Mn absorption which could partially explain increased Ca availability in soil. However, the lack of Ca change in aboveground tissues indicates the need for more research to elucidate the effects of residue application on Mn and Ca dynamics in *P. taeda* plantations.

Concentrations of Mn in needles ( $190 \text{ mg kg}^{-1}$ ) and bark ( $26 \text{ mg kg}^{-1}$ ) may be related to increased average annual increment in *P. taeda*. This information indicates that bark could be used as an alternate tissue for nutrition diagnosis, which makes tissue collection easier. Other alternatives may be useful for other nutrient assessments; for example, root tissue has been proposed for P evaluations since collecting needles can be difficult, especially in older trees. High Mn values in needles tissue is not unusual (Consalter et al. 2020; Sass et al. 2020). In addition, K and Mg concentrations

were close or slightly below critical levels ( $3.5 \text{ g kg}^{-1}$  and  $0.6 \text{ g kg}^{-1}$ , respectively) recognized for needle tissue (Pauletti and Motta 2018; Albaugh et al., 2008). High Mn levels could be related to nutritional imbalance rather than soil acidity based on evidence observed in the field, in control plot trees, and in areas adjacent to the experimental site.

In addition to Mn, decreases in Fe, Al, and B were observed in live branch and needle tissues. Decrease in P, Mg, and S observed in live branches (along with decreased P and Ca in needles) were in contrast to increases observed for soil P and Ca. This may have occurred due to increased tree growth without changing nutrient amount, consequently reducing nutrient concentrations (dilution effect). Likewise, Sass et al. (2020) reported P dilution in needles from the same forest site 3 years after residue application. It is important to note that dilution can result in P concentrations well below the critical level of  $1.2 \text{ g kg}^{-1}$  (Albaugh et al. 2010).

Therefore, the decrease in Mn and Al levels may have resolved a possible Mn toxicity or antagonistic effect interfering with absorption of other nutrients, thus increasing growth. Many studies have reported that high levels of exchangeable soil Al did not impact growth of *Eucalyptus* and *Pinus* species, indicating that both species are tolerant to this element (Gomes et al. 2019; Bassaco et al. 2018). The high values of macronutrients seen with the addition of  $49 \text{ T ha}^{-1}$  of residue were a result of better development and general tree growth with this rate of residue application.

## Forest biomass nutrient content

Carbon stored in wood and bark was interesting since the concentration of this element in these fractions did not change (Table 5). Our work showed an increase of C in wood and bark of  $\sim 20 \text{ T ha}^{-1}$  with the application of industrial residue (Table S4). Conditions favorable to improved soil nutrition can increase tree canopy growth and consequent accumulation of C (Samuelson et al. 2004; Tumushime et al. 2019). While Jokela et al. (2010) suggested that growth gains and C accumulation are greater in low fertility areas, Tumushime et al. (2019) reported that responses to fertilizer were lower in more productive areas.

Other nutrients usually tend to follow the accumulation pattern of biomass and C (Vogel et al., 2011). In our work, only N, K, Ca, Mg, and S in wood were affected by residue application (Table S4). This change was expected due to increases in the stand (Angel et al. 2019), however changes in other tissues and nutrients were also expected (Zhao et al. 2014).

Relating aerial biomass to nutrient content, work with 16 year-old (Sixel et al. 2015) and 25 year-old (Tumushime et al. 2019) *P. taeda* found that the needle fraction was the second most accumulator of aerial biomass behind wood tissue, which was different from our work that found an order of wood, bark, and live branches before needles. Although older forests tend to have higher amounts of nutrients than immature forests (Albaugh et al. 2004), P in wood shows little variation over time. We found  $12.8 \text{ kg ha}^{-1}$  of P (7 year-old), while Tumushime et al. (2019) found an average of  $13.5 \text{ kg ha}^{-1}$  (25 year-old), and Moro (2008) estimated  $16\text{--}11 \text{ kg ha}^{-1}$  for 8, 12, 16 and 20 year-old forests. Greater P variations are found in needles due to age (Sixel et al. 2015; Tumushime et al. 2019). Thus, adequate diagnosis and nutritional management of sites may require nutrient monitoring in different forest compartments over time.

## Litter

Total necromass found in this study (from 28.9 to 52.0 T ha<sup>-1</sup>) was smaller than observations by Trevisan et al. (1987) under similar conditions of soil and climate; their values ranged from 43.0 to 83.9 T ha<sup>-1</sup> for 17 year-old *P. taeda* on three growth sites (poor, medium, and high). Differences between studies could be related to the age of our system at sampling (i.e., 7 year-old trees). In another Brazilian study, Rabel et al. (2020) found variation between 35.8 to 42.1 T ha<sup>-1</sup> for 13 year-old *P. taeda* that received cellulose residue applications.

Results from our study confirms a high potential for litter accumulation in this area. For *P. taeda* systems, high potential for necromass accumulation on forest floors has been related to poor soil fertility and poor growth sites (Reissmann and Wisniewski 2015). Although results from the control indicate necromass accumulation potential, these results are inconsistent with increases in both forest floor necromass and tree growth as was observed in our study. Thus, more work is need to further evaluate the effect of fertility and fertilization on necromass accumulation on forest floors.

The observed trend for new FF accumulation as a result of residue application suggests some enhancement of litterfall deposition. Increases in old FF could be related to tree growth as well as possible addition of charcoal from residue application. Charcoal has been shown to have a long life span and has been observed in Amazonian dark earths (*Terra Preta de Índio—TPI*) on pre-Columbian Amerindian sites and near charcoal kiln production sites in Europe (Mastrolonardo et al. 2019; Hardy et al. 2017). Despite low change in soil properties, residue applications possibly influenced decomposition processes while adding recalcitrant C in the form of charcoal. Enhanced humification rate as proposed by Rabel et al (2020) may help explain FF increases from residue application. Our results confirmed higher level of necromass maintenance with addition of nutrients in the form of residue (Rabel et al., 2020) and lime/fertilizer (Consalter 2018). In contrast, Marschner and Wilczynski (1991) and Lundström et al. (2003) reported a large reduction in FF necromass from applying alkaline amendments (lime and ash), while Jandl et al. (2003) observed similar effects from a fertilizer/lime combination in European forests.

The variation in C for new FF (437 to 460 g kg<sup>-1</sup>) was very close to the range observed in needles (436 to 468 g kg<sup>-1</sup>—Table 4) which may reflect new litterfall input. Our C variation was larger than values reported by Marschner and Wilczynski (1991) for FF from limed (167 g kg<sup>-1</sup>) and control (267 g kg<sup>-1</sup>) forest plots. Prior to accounting for contaminants, these authors had corresponding values of 513 and 484 g kg<sup>-1</sup> in these respective treatments. This illustrates that our new FF appears to represent the FF fraction with minimum disturbance and mineral contamination.

Despite our efforts towards extracting mineral contaminants, the reduction in C concentration from new FF to old FF clearly indicated a dilution from mineral contaminants as reported by Marschner and Wilczynski (1991). This dilution effect could explain the wide variation of C concentration found by Koschke et al. (2011); these authors evaluated 38 sites in Germany for the influence of black carbon and reported ranges of 230 to 484 g kg<sup>-1</sup> and 66 to 462 g kg<sup>-1</sup> for partially and highly decomposed FF, respectively. Mineral contamination of FF seems to be greatly influenced by litter age since Rabel et al. (2020) reported ash percentages ranging from 2.5% for fresh litter to more than 50% for the fine humidified FF fraction.

Increased litter due to residue application can be important for soil conservation due to the undulating landscape and sandy soil texture of the study region. Additionally,

more than  $7 \text{ T ha}^{-1}$  of C was accumulated in litter at the residue rate that provided maximum growth ( $49 \text{ T ha}^{-1}$ ) relative to the control. Drum et al. (2019) confirmed increased C sequestration in aboveground tissues and litter compartments as result of improved *Pinus* forest management.

## Conclusions

Regardless of rate, *P. taeda* timber volume was greatly increased by industrial residue application, which suggests a high potential for yield enhancement. Since tree height was not influenced by residue application, increases were related to diameter improvements. Despite these improvements, there was no difference in the proportion of tree biomass components (wood, bark, needles, and live and dead branches). A possible direct relationship between enhanced tree growth and total litter mass suggests increased litterfall input. Fragmented and humified forest floor litter fractions became darker with increased residue rate, indicating that these fractions trap charcoal fragments associated with industrial residues. In contrast to tree growth, only a few soil property and tissue nutrient parameters showed slight changes, illustrating the difficulty of associating *Pinus* growth with these factors. Changes in soil properties were restricted to increased P and Ca availability, however decreases in these nutrients were observed in tree tissues, likely due to a dilution effect. Although no change in soil pH was observed in our study, the noted decreases in needle Mn, Fe, B, and Al would normally be related to increased soil pH. While excess Mn could compromise tree growth under acidic conditions (due to toxicity or imbalance with other nutrients), residue application was observed to decrease Mn in all evaluated tissues. Wood quality needed for production processes was not impacted since wood density remained within industry standards regardless of applied residue rate. Application of industrial residues to low fertility soils planted with *P. taeda* is recommended for accelerated growth and stand production.

**Supplementary Information** The online version contains supplementary material available at <https://doi.org/10.1007/s11056-021-09902-w>.

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