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Mineral composition of eucalyptus bark and wood as a function of sample height and gypsum application

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Abstract: Whether gypsum application changes mineral composition of *Eucalyptus urograndis* bark and wood along a tree height gradient is uncertain. Therefore, an experiment was established to examine three rates of broadcasted gypsum (1.2, 4.8, and 9.6 Mg ha^{-1}; control); applied before planting. Three years after application, discs were collected to obtain bark and wood chemical composition from the tree base and at 25, 50, 75, and 100% of total height. Results shows that calcium (Ca), iron (Fe), aluminum (Al), and manganese (Mn) can accumulate in wood at the tree base, while potassium (K), sulfur (S), and phosphorus (P) tend to accumulate in the upper portions of trees. Gypsum application increased concentrations of Ca, S, and P in wood; Ca, S, P and copper (Cu) in bark; and decreased Mn in bark and wood. Findings suggest that high gypsum rates increased exportation of Ca, S and P by wood. Additionally, to accurately estimate nutrient exportation it is important to sample at multiple height positions along the tree trunk.
Keywords: *Eucalyptus urograndis*; forest; nutrition; elemental composition; sampling.

1. Introduction

Occupying 5.67 million planted hectares in 2016, the genus *Eucalyptus* is the most cultivated forest species in Brazil, generates 1.1% of the national wealth, and constitutes 6.2 % of the industrial gross domestic product (Ibá, 2017). The success of eucalyptus is linked to fast growth, short cultivation cycles (compared to other forest species), and high yields.

Major soils cultivated with eucalyptus are of low fertility (Foelkel, 2005). The tropical/subtropical climate of Brazil favors intense weathering that results in soils with toxic concentrations of aluminum (Al), low pH, and low calcium (Ca) availability. This issue is very important since Ca is one of the most abundant nutrients found in eucalyptus trees (Guimaraes et al., 2015; Viera et al., 2015), and Ca reallocation is necessary to prevent decreased productivity (Santana et al., 2002).

Although eucalyptus bark represents a small portion of commercial logs compared to wood, 70 to 80% of total Ca was found in bark, while 30% was in wood tissue (Santana *et. al.*, 2008; Macana, 2017; Schumacher et al., 2001). Thus, harvest systems that remove only wood is more sustainable since less Ca is removed from forests. Compared to wood, bark also had higher concentrations of phosphorus (P), potassium (K), and magnesium (Mg) (Guimaraes et al., 2015). These facts are important in understanding nutrient exportation due to harvest activities.

Limestone ($CaCO_3$ and $MgCO_3$) and gypsum ($CaSO_4$) are low-cost Ca sources in Brazilian agriculture. Gypsum is a byproduct from phosphate fertilizer production, contains approximately 20-24 % Ca, and also contains 15-19 % sulfur (S) (Korcak, 1998). In addition to supplying Ca and S, gypsum can act as a soil conditioner by reducing Al toxicity when used in high amounts (Sodré et al., 2001, Ernani et al., 2001). Despite potential benefits of gypsum application on soil chemical properties, eucalyptus growth responses have varied from large increases (Rodrigues et al., 2016), little or no increases (Macana, 2017), and decreases in some instances (Gabriel et al., 2018).

When applied in large amounts, changes associated with gypsum used as a soil conditioner may reach lower soil layers and result in nutrient leaching (Caires et al., 2006; Ramos et al., 2013; Leite et al., 2007, Soratto and Crusiol, 2008). These changes can induce root growth and distribution changes (Zandoná et al., 2015), which can influence volume of soil exploration and overall plant nutrition. However, variable gypsum effects on eucalyptus roots have resulted in no response or decrease (Macana, 2017) or positive responses (Rodrigues et al., 2016).

Changes in soil attributes and root growth from gypsum application could influence tree nutrition. Under controlled conditions, Christo and Santos (1990) observed increased Ca and S in eucalyptus seedling tissue with gypsum application. Under field conditions, Macana (2017) found increases in foliar Ca and S concentrations of eucalyptus 6 months after gypsum application compared to the control; however, this effect was not maintained after 30 months. Rodrigues et al. (2016) observed foliar increases in Ca and S with lime and gypsum applications compared to only lime, but only in leaves from the middle third of the crown for 18-month-old eucalyptus trees. These findings suggest that Ca and S applied via gypsum seemed to influence plant tissue concentration but can vary with plant age and tissue type.

The influence of gypsum on other nutrient levels has been investigated: Christo and Santos (1990) reported reduced K, Macana (2017) observed increased K and reduced Mg, and Rodrigues et al. (2016) only found reduced Mg. These results suggested that it is important to evaluate how gypsum applications (Ca and S) influence other nutrient levels to better assess overall gypsum effects.

Although foliar tissue is preferentially used as a plant diagnostic tool, bark composition was an effective means of evaluating manganese (Mn) toxicity in eucalyptus (Leite et al., 2014). The nutrients in the Pinus taeda wood tissue also showed a good indicator to change provided by residue amendment (Rodriguez et al., 2020; Pereira et al., 2022) or variation in soil fertility (Ercole et al., 2024, Marques et al., 2024; Rodrigues et al., 2025).

To improve nutrient export predictions and to establish a sampling methodology for this determination, ascertaining nutrient variation along tree height is necessary (Rytter, 2002; Sander and Ericsson, 1998). It is expected that mobile elements concentrate in upper portions of trees, while the opposite occurs for less mobile elements. Field samples normally collected at breast height (~1.3 m) can over- or underestimate the amount of nutrients in bark and wood. Thus, the aim of the present study was to assess macro- and micronutrients in wood and bark of *Eucalyptus urograndis* (*E. urophylla* x *E. grandis*) at different sampling heights as a function of gypsum application.

2. Material and Methods

The experiment was installed in the municipality of Jaguariaíva, Paraná, Brazil (24°15’04” S latitude, 49°42’21” W longitude, 850 m elevation). Based on Köppen classification, the regional climate is Cfb with a temperate moist mesotherm. Monthly average temperatures were less than 22ºC and average annual rainfall was 1400 to 1600 mm and was well distributed (Alvares et al., 2013). Soil was formed from sedimentary rock of the Furnas formation (Paleozoic era) and was classified as a dystrophic Oxisol with sandy-loam texture (SOLOS, 2018).

The area had previously been used for pine cultivation. Before our study initiation, 20 soil samples were collected using a Dutch auger and mixed to obtain composite samples for the 0-10, 10-20, and 20-40 cm soil depths. All samples were air dried, homogenized, and passed through a 2 mm mesh sieve. Samples were analyzed for pH ($CaCl_2$ and SMP), Ca^{2+}, Mg^{2+}, Al^{3+} (extracted with KCl 1mol L^{-1}), K^+, P (extracted by Mehlich I), and organic carbon (C) by colorimetry according Embrapa (1997). Determination of Al^{3+} was done by titration with NaOH, exchangeable Ca^{2+} and Mg^{2+} by atomic absorption, K^+ by flame photometry, and P by colorimetry. Based on previous work (Bassaco et al., 2018), N was not determined due to no influence of N on Eucalyptus growth in this specific experiment. Soil chemical characteristics are shown in Table 1.

A randomized block experiment with four gypsum application rates (0, 1.2, 4.8, and 9.6 Mg ha^{-1}) and four replications was used. Each plot measured 24 m x 24 m. There was a total of 64 trees per plot, planted on 3 m x 3 m spacings. The chemical composition of utilized gypsum was 19.3% Ca and 15.83 % S.

TABLE 1 – Soil chemical properties before study initiation at the municipality of Jaguariaíva, Paraná, Brazil.

Depth (cm)	pH $CaCl_2$	Al^{3+}	H+Al	Ca^{2+}	Mg^{2+}	K^+	BS[1]	CEC pH 7	P	C_{org}	V	m
		------------------------- $cmol_c$ dm^{-3}						-----------------------	mg dm^{-3}	g dm^{-3}	--- %	---
0–10	3.9	0.9	6.7	0.5	0.3	0.03	0.83	7.53	3.5	12.4	11	52
10–20	3.8	1.0	7.2	0.2	0.1	0.04	0.34	7.54	0.9	8.7	4.5	75
20–40	3.9	1.0	5.8	0.1	0.1	0.03	0.23	6.03	0.4	8.7	3.8	81

[1]BS=Sum of bases (Ca + Mg + K), CEC= Cation exchange capacity, C_{org}= organic carbon, V= Base saturation, and m= Aluminum saturation

3. Results and Discussion

3.1. Effect of gypsum application on bark nutrient and Al concentrations

Gypsum application resulted in changed Ca, S, P, Mn, and Cu contents in bark; this occurred in at least one of the five sample heights. Effects on Al, Mg, K, Fe, and Zn ($p>0.05$) were not observed (Figure 1).

In general, Ca concentration increased from the tree base to 25 % tree height then decrease progressively. At the highest gypsum rate, Ca concentration decreased from the base to top of the tree suggesting a change in distribution under high Ca supply. Gypsum applied at the maximum rate significantly increased Ca concentration ($p=0.01$) at the tree base and at 50 % tree height (Figure 1A). These results differ from those reported by Macana (2017) who did not find an increase in bark Ca concentration with Ca applications of 442 kg ha^{-1} as gypsum and limestone. However, findings indicated that high amounts of Ca were necessary to obtain concentration increases in bark; only the two largest gypsum rates resulted in Ca concentrations higher than the control at 50 % of tree height.

In this study, obtained contents of Ca in bark (2.77 to 45.13 g kg^{-1}, average of 15.93 g kg^{-1}) were greater than the findings of Rodrigues et al. (2016) for 18-month-old trees (4.8, 5.2, 7.2, and 7.9 g kg^{-1} for fertilizer [N and P], reactive natural phosphate, Lime, and Lime/gypsum, respectively) and greater than findings (11.4 g kg^{-1}) reported by Viera et al. (2013B) for 6-month-old trees co-associated with corn. Viera et al. (2015) reported higher contents (22.65 g kg^{-1}) for 10-year-old trees. However, the Ca contents found in the present work were similar to the predicted value of 17.6 g kg^{-1} for bark of 2- to 4-year-old trees based on a eucalyptus survey in Brazil (Santana et al. 2000).

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Gypsum applied at the highest rate also increased bark S concentration (Figure 1 E). The 4.8 Mg ha^{-1} rate also enhanced S concentration ($p<0.01$) except at the tree base (0% tree height). Similar to Ca, a high rate of S in the form of gypsum was necessary to influence bark S concentration. The level of S reported by Viera et al. (2013A) was 0.35 g kg^{-1}, which was higher than the 0.15 to 0.31 g kg^{-1} obtained in our study. The influence of gypsum application on Ca and S was expected since they are major constituents of gypsum and high rates can enhance soil availability.

Among other macronutrients evaluated, only P was affected by gypsum application. Increased P at 25% of tree height was observed ($p=0.01$) (Figure 1D). Contents of P in this study (0.3 to 0.9 g kg^{-1}) were very close to the 0.6 g kg^{-1} reported by Guimaraes et al. (2015).

Our findings did not confirm the negative effects of gypsum on foliar nutrient concentrations as described by Macana (2017) and Rodrigues et al. (2016) for Mg or for K reported by Christo and Santos (1990). Our Mg levels were close to the 1.9 and 2.0 g kg^{-1} reported by Rodrigues et al. (2016) and Santana et al. (2000), respectively. For K, our levels were below the 6.28 g kg^{-1} reported by Viera et al. (2013A).

Among micronutrients, Mn was clearly most affected by gypsum application. The Mn concentration decreased at the highest gypsum application rate for four of the five tree heights evaluated compared to the control ($p<0.01$, $p=0.02$, $p=0.02$, $p=0.03$, respectively for 25, 50, 75, and 100 % of tree height). The capacity of Ca to decrease Mn toxicity is well known (El-Jaoual and Cox, 1998). The level of Mn obtained in our work varied between 188 and 790 mg kg^{-1} and were lower than the 1813 mg kg^{-1} of Viera et al. (2013A). Leite et al. (2014) reported Mn values between 468 and 889 mg kg^{-1} for trees with low occurrence of Mn toxicity symptoms and between 1899 and 2734 mg kg^{-1} for trees with high occurrence of Mn toxicity symptoms. Thus, it is unlikely that our contents represent Mn toxicity. Although unclear, decreased Mn from gypsum application could be problematic since concentrations ranged from levels considered deficient to mildly toxic.

Gypsum application also changed Cu levels at the tree base ($p=0.03$), where the highest rate was different from the control (Figure 1J). In our work, Fe levels varied between 36 and 151 mg kg^{-1} and were greater than the 30.27 mg kg^{-1}reported by Viera et al. (2013B). Although gypsum has been reported to diminish Al toxicity (Gerárd, 2016), there was no indication a decrease from gypsum application (Figure 1G).

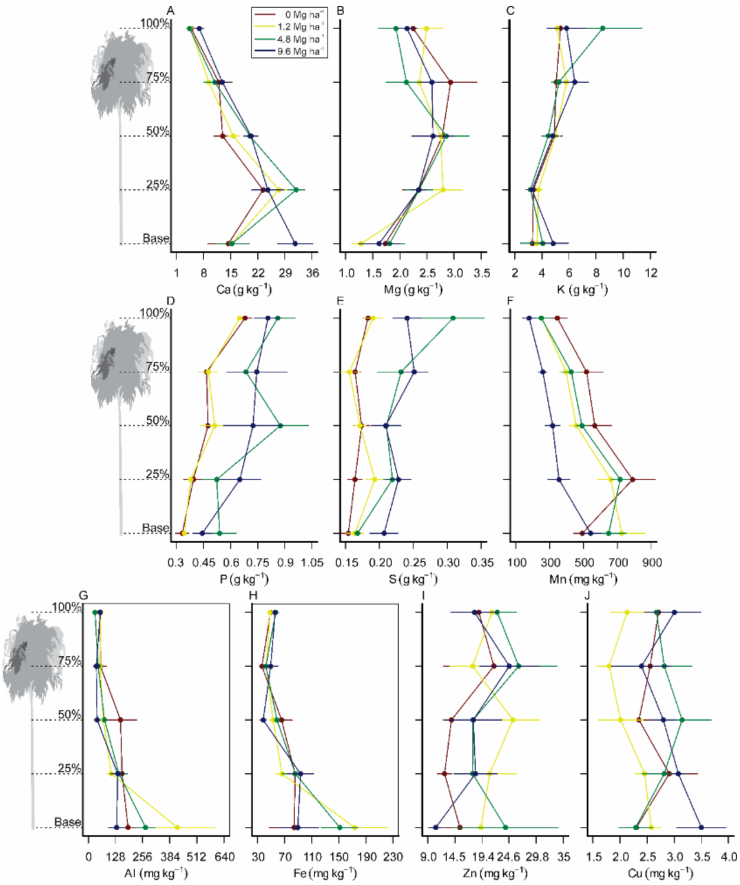

Figure 1 – Elemental composition of bark (average and standard error) at different sampling heights of 3-year-old hybrid *Eucalyptus urograndis* trees (n= 4) receiving gypsum applications of 0, 1.2, 4.8, and 9.6 Mg ha^{-1} at the municipality of Jaguariaíva, Paraná, Brazil. Alphabetical labeling of graphs represent: Ca, Mg, K, P, S, Mn, Al, Fe, Zn, and Cu, respectively.

3.2. Effect of relative height on bark concentrations

An influence of height sampling on bark levels was observed for all nutrients ($p>0.05$) except Cu and Zn. These effects are clearly shown in Figure 1. For the control, there was difference in levels of Ca ($p=0.02$), K ($p=0.04$), and P ($p<0.01$) in relation to height. For the lowest gypsum rate, there were differences in Ca ($p<0.01$), Mg ($p=0.01$), K ($p=0.01$), P ($p<0.01$), Mn ($p<0.01$), Al ($p=0.03$), and Fe ($p<0.01$). When applied at 4.8 Mg ha^{-1} (intermediate gypsum rate), effects were observed for Ca ($p<0.01$), P ($p=0.02$), Al ($p<0.01$), Fe ($p<0.01$), Mn ($p<0.01$), and S ($p=0.04$). However, the highest gypsum rate affected only Ca ($p<0.01$), Al ($p=0.02$), and Mn ($p<0.01$).

The behavior of Ca regarding to height sampling was similar among the control and gypsum rates of 1.2 and 4.8 Mg ha^{-1}. For these treatments, Ca increased from the tree base to 25 % of tree height and decreased in upper portions of the tree. These results highlight that increased Ca at the tree base occurred only for the highest gypsum rate (Figure 1A).

Similar to Ca, decreased levels according to height sampling were also observed for Mn, Al, and Fe (Figure 1F-H). However, an inverse relationship was observed for K, P, and S where levels increased as sampling height increased (Figure 1C-E).

As mentioned above, elements with high mobility or high redistribution capacity generally concentrated in new tissues while those with low mobility concentrated in old tissues. The low mobility of Mn in bark was confirmed by Leite et al. (2014) who assessed areas affected by Mn toxicity. These authors observed greater values in bark on the lower third of the tree trunk with no effect in wood tissue. The low mobility of Ca was also reported by Rodrigues et al. (2016) who found contents up to ten times smaller in leaf tissues from the upper third of the canopy compared to the lower third.

3.3. Effect of gypsum application on wood concentrations

Although the effects of gypsum on wood concentrations of Ca, S, and P were the same as those observed for bark, changes were noticed in levels of Al and Zn (Figure 2 A, E, D, G, I). Unlike observations for bark, effects on Cu and Mn were not observed in wood tissue ($p>0.05$). In regards to Mn levels in wood, values reported by Viera et al. (2013A) were approximately ten times greater than the range observed in our study (12.72 to 36.04 mg kg^{-1}).

The highest gypsum application rate led to the largest Ca increase in wood at the tree base ($p<0.01$), and at 25% ($p=0.04$), 50% ($p=0.01$), and 100% ($p=0.03$) of tree height. The highest rate led to larger values than the control and 1.2 Mg ha^{-1} treatments at the 25% height sampling, while the 4.8 Mg ha^{-1} treatment led to larger contents for the tree base, and the 50 and 100% height samplings. Our Ca range (0.5 to 2 g kg^{-1}) was lower than findings of Rodrigues et al. (2016) and Viera et al. (2013A), who reported concentrations of 3.02 and 1.42 g kg^{-1}, respectively. Sulfur concentrations changed with gypsum at the 50 % ($p=0.04$) and 100 % ($p=0.03$) height samplings; the 4.8 Mg ha^{-1} rate resulted in higher contents than the maximum rate and control.

The highest values of Al and Zn were obtained with the 1.2 Mg ha^{-1} treatment. For Al levels, in the relative height sampling of 25 %, the treatment of 1.2 Mg ha^{-1} differed from other treatments including the control ($p=0.03$). A decrease from 320 to 32.3 mg kg^{-1} was observed from the base of the tree to 25% of the height for the control treatment, respectively. Regarding to Al, wood levels were not expected to increase since gypsum can act as a soil conditioner that neutralizes Al via Ca^{2+} reactions that decrease Al^{3+} in soil solution and consequently its availability (Raij, 2013; Macana, 2017). For Zn levels at 100 % of relative height, the rate of 1.2 Mg ha^{-1} resulted in higher amounts of this element compared to the control and 9.6 Mg ha^{-1} ($p=0.01$). Phosphorus was also different at 100 % of relative height ($p=0.02$), with the 4.8 Mg ha^{-1} treatment having lower concentrations in comparison to other rates.

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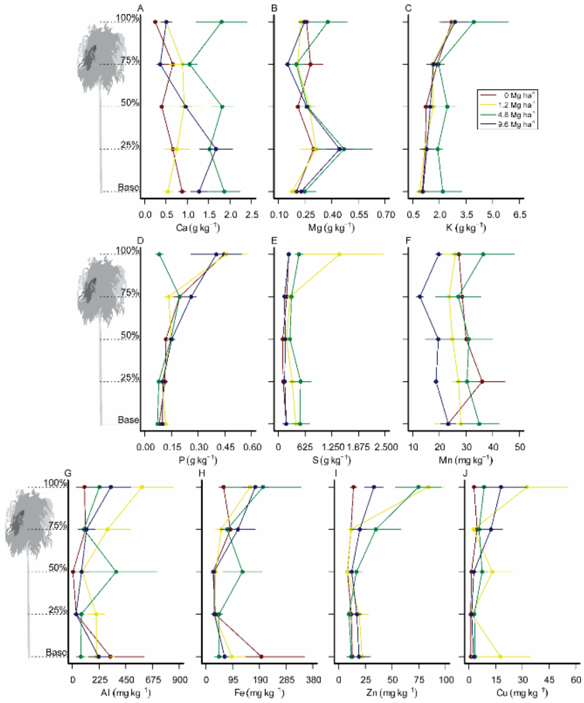

Figure 2 – Elemental composition of wood (average and standard error) at different sampling heights of 3-year-old hybrid *Eucalyptus urograndis* trees (n= 4) receiving gypsum applications of 0, 1.2, 4.8, and 9.6 Mg ha^{-1} at the municipality of Jaguariaíva, Paraná, Brazil. Alphabetical labeling of graphs represent Ca, Mg, K, P, S, Mn, Al, Fe, Zn, and Cu, respectively.

3.4. Effect of relative height on wood concentrations

Differences due to height sampling were observed for Ca, Mg, K, P, and Zn levels in wood, but had no effect on S, Al, Cu, and Mn ($p>0.05$). Calcium was different in the control with greater levels at the tree base ($p=0.03$) in comparison to 50 and 100 % relative height. The highest rate of gypsum, it was observed the higher concentration of Ca at 25% relative height compared to 75 and 100 % relative heights ($p<0.01$). Potassium was affected in the control ($p<0.01$), with greater concentrations at 100 % when compared to the tree base, 25, and 50 % relative heights. For the 1.2 Mg ha^{-1} treatment at 100 % relative height, K concentration was higher than for other rates ($p<0.01$). The same was observed at the highest rate ($p<0.01$). Thus, the 4.8 Mg ha^{-1} rate was the only treatment that was shown not to be affected by sampling height. In general, K accumulation was similar from the tree base to 75 % relative height but increased with greater height.

Effects on P were observed for all treatments. For the control, P levels were greater at 100 % relative height ($p<0.01$), and the 75 % relative height was different from the tree base and 25 % relative height. For the 1.2 Mg ha^{-1} rate, only the 100 % relative height was different compared to other samplings ($p<0.01$). For the 4.8 Mg ha^{-1} rate, the 75 % relative height was different from all others, while the 50 % relative height was different from the tree base, and 25% was different from 100 % relative height ($p<0.01$). For the highest rate, P levels at 100 % relative height were different from the tree base, 25% and 50 % relative heights ($p=0.02$). In general, P levels increased with sampling height, which was similar to that observed in bark.

More Mg accumulated at the 25% relative height and decreased towards extremities ($p<0.01$). Iron level was affected by the 1.2 Mg ha^{-1} application rate ($p=0.02$), with differences between 100% and 25 % relative heights and between 50% and 75 % relative heights. Effects from this rate were also observed for Zn, with higher amounts noticed at 100% in comparison to other relative heights ($p<0.01$). In addition, the 4.8 Mg ha^{-1} rate ($p=0.04$) also resulted in greater Zn levels at 75 % relative height.

3.5. Relationship among elemental levels in bark and wood

It was observed greater elemental accumulation in the bark in the following order: Ca>K>Mg>P>Mn>S>Al>Fe>Zn>Cu. At all relative sampling heights, Ca, K, Mg, P, and Mn levels were greater in bark than in wood tissue ($p<0.01$ for both). In support of our findings, other studies have reported high values for Ca, Mg, K, and P in bark for different soils and climatic conditions (Santana, 2000; Leite, 2001; Barbosa et al., 2014; Guimaraes, 2015). Greater Mn values in bark vs wood were reported by other authors (Leite, 2001; Leite et al., 2014; Guimaraes, 2015; Dick et al., 2017).

In general, Ca and Mn values were 15 to 20 times greater in bark than in wood. Bark also contained 10 times more Mg, 2.5 times more K, and 4 times more P. This is very important since adoption of harvest practices that maintain bark in the field could significantly decrease exportation of these nutrients. The bark to wood ratios of Ca and Mg were greater in our study compared to those reported by Macana (2017); there was 5 times more Ca and 2 times more Mg in bark than in wood. In their work, an application of 620 kg ha^{-1} CaO (dolomitic lime and gypsum) resulted in no change.

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Wood tissue followed a different accumulation sequence where: K>Ca>Mg>P>S>Al>Fe>Mn>Zn>Cu. This sequence highlights that K accumulation in wood increased while Mn decreased relative to bark. Although not reported in the literature, our results show that Al is an important constituent in both wood and bark; levels were noted to be slightly higher in wood. Although the use of gypsum has been related to reducing Al uptake, we observed a limited effect of gypsum for eucalyptus in our study.

Levels of Fe, Zn, Cu, and S were somewhat similar in bark and wood, with values being slightly greater in wood. Values of these elements reported in the literature were very close or slightly greater than our observations (Leite, 2001; Guimaraes et al., 2015; Dick et al., 2017).

4. Conclusion

Application of gypsum increased Ca and S levels in tree trunks. Calcium level was higher in bark than wood and decreased from the tree base to the top of the tree. In contrast, S levels were higher in wood and increased from the tree base to the top of the tree. Bark levels of P, Cu, and Mn were also affected by gypsum application. Concentrations of P and Cu increased with gypsum application, while Mn decreased. The concentration of Mn also decreased from the tree base to the top of the tree. Concentrations of P, Zn, and Al in wood were impacted by gypsum application. Gypsum decreased P in the upper portion of the tree trunk, but increased Zn and Al. Accumulation of Al was greater in wood than bark. Overall, large amounts of gypsum were required to promote changes in the elemental composition of eucalyptus bark and wood. In general, high mobility elements were concentrated in upper portions of the tree trunk, while those with low mobility concentrated closer to the tree base. Thus, multiple samplings at different tree heights are needed to accurately estimate nutrients exported as wood and bark during harvests.

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