

**Gypsum effects on eucalyptus nutrition in subtropical Brazil****Efeitos da aplicação de gesso na nutrição de eucalipto no Brasil subtropical**

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**ABSTRACT**

Agricultural gypsum has been used to mitigate soil Al toxicity and increase Ca and S under low availability conditions. To determine the influence of gypsum application on the nutritional status of *Eucalyptus urograndis*, a study was established on low fertility acid soil at two sites (Ventania and Jaguariaíva) under subtropical conditions in southern Brazil. The experiment consisted of a control and six rates of agricultural gypsum (i.e., 0, 0.3, 0.6, 1.2, 2.4, 4.8, and 9.6 T ha<sup>-1</sup>) broadcast on the soil surface during seedling transplanting. Foliar tissues collected at 12, 24, and 36 months after transplanting were evaluated for nutrients (Ca, Mg, K, P, S, Fe, Mn, Zn, Cu, B) and other elements (Al, Pb, Cr, Cd, Ba, As). Despite similar soil properties at the two experimental sites, nutritional effects and site responses were variable with plant age. At Jaguariaíva, gypsum amendment enhanced Ca concentration by 2-fold at 12 months and 55 % at 24 months but had no effect at 36 months. Gypsum increased S concentration in the first year at Jaguariaíva for the highest rate and in all three years at Ventania for the two highest rates. Gypsum use decreased Mg tissue concentration for both sites but at different time scales. The Ventania site exhibited diminishing Al and Mn and increasing P in foliage over time. Findings suggest that agricultural gypsum applied to young eucalyptus stands in subtropical Brazil will have little effect on foliar elements in the long-term.

**Keywords:** soil amendment, nutrients, potentially toxic elements, calcium sulfate, cultivated forest.

**RESUMO**

O gesso agrícola tem sido utilizado para mitigar a toxicidade do Al no solo e aumentar os teores de Ca e S em condições de baixa disponibilidade. Para determinar a influência da aplicação de gesso no estado nutricional de *Eucalyptus urograndis*, foi realizado um estudo em solos ácidos com baixa fertilidade em dois locais (Ventania e Jaguariaíva - PR) em condições subtropicais do Brasil. O experimento consistiu em um tratamento controle e seis taxas de gesso agrícola (0,3; 0,6; 1,2; 2,4; 4,8; e 9,6 T ha<sup>-1</sup>) aplicadas na superfície do solo durante o transplante de mudas. Os tecidos foliares coletados aos 12, 24 e 36 meses após o transplante foram avaliados quanto aos teores de nutrientes (Ca, Mg, K, P, S, Fe, Mn, Zn, Cu e B) e outros elementos (Al, Pb, Cr, Cd, Ba e As). Apesar das propriedades similares do solo nos dois locais experimentais, os efeitos nutricionais e as respostas dos locais foram variáveis com a idade da planta. Em Jaguariaíva, o uso de gesso aumentou a concentração de Ca em duas vezes aos 12 meses, e 55% aos 24 meses da planta, mas não teve efeito aos 36 meses. As doses mais altas de gesso aumentaram a concentração de S no primeiro ano em Jaguariaíva e nos três anos em Ventania. O gesso diminuiu a concentração de Mg no tecido vegetal nos dois locais, mas em escalas de tempo diferentes. Ventania apresentou teores decrescentes de Al e Mn, e crescentes de P nas acículas ao longo do tempo. Os resultados sugerem que o gesso agrícola aplicado a plantios novos de eucalipto no Brasil subtropical terá pouco efeito sobre os elementos foliares a longo prazo.

**Palavras-chave:** condicionante do solo; nutrientes; elementos potencialmente tóxicos; sulfato de cálcio; floresta plantada.

## 1 INTRODUCTION

Planted forests correspond to 7% of total forested area worldwide and are composed of trees established through planting and/or deliberate seeding of native or introduced species. (FAO, 2010). A substantial proportion of recently established planted forest, especially in the tropics and subtropics, includes eucalyptus (Binkley et al., 2017; Brockerhoff et al., 2013). In 2000, eucalyptus plantations occupied ~17 million ha (FAO, 2000); by 2009, this area was ~20 million ha (Iglesias-Trabado and Wilstermann, 2009). This steady increase in planted forest occurred primarily between 35°S and 35°N (Ferreira et al., 2018). The FAO (2010) predicts that planted forests will increasingly contribute to the global supply of wood, fiber, and fuel thereby reducing pressure on natural forests.

Plantations using suitable eucalyptus species in tropical and subtropical regions of Brazil are among the most productive forests worldwide; development of these Brazilian forests resulted in a 4-fold increase in wood productivity between 1970 to 2015 (Binkley et al., 2017). More recent assessments indicate that eucalyptus occupies over 7 million ha in Brazil (IBGE, 2017). Minas Gerais, Mato Grosso do Sul, São Paulo, Paraná, and Rio Grande do Sul are the main producing states in Brazil and represent 70% of the planted area. This wide distribution reveals the effect of different physiographic, edaphic, and climatic conditions on eucalyptus growth, with productivities varying from 15 to 107 m<sup>3</sup> ha<sup>-1</sup> yr<sup>-1</sup> (Santana et al., 2002).

Among other factors, sustainability of forest ecosystems is associated with nutrient balance stability in the short, medium, and long term (Santana et al., 2002). Nutrient utilization efficiency may vary by species, genotypic functional differences, genotypic-environmental interactions, and soil nutrient availability. In general, nutrient utilization efficiency varies for each nutrient and tends to increase under low availability conditions (Santana et al., 2002).

Nutritional support from forest residues (i.e., litter layer) has a crucial effect on yield (Rocha et al., 2018). After planting seedlings, the soil is no longer disturbed resulting in a “no-tillage” system that is prone to chemical stratification, resulting in high pH and base levels in the upper layers of the soil profile. On the other hand, low amounts of base cations (especially Ca) and Al toxicity in the subsoil impacts root growth and restricts access to water and nutrients. This issue is critical in Brazil since exchangeable Al is found in most cultivated soils throughout the country (Caires and Guimarães, 2018).

Amending soils with gypsum to improve physical and chemical properties is increasingly being practiced. Surface application of gypsum was found to improve water infiltration, increase soil water content, and correct subsoil Al toxicity (Kost et al.,

2018). When applied to the soil surface, gypsum moves into the soil profile during drainage; this increases  $\text{Ca}^{2+}$  supply and reduces toxic levels of  $\text{Al}^{3+}$ , especially in subsurface layers. As a result, root growth and absorption of water and nutrients improve. Since root proliferation can be limited in soils with very high  $\text{Al}^{3+}$  and low  $\text{Ca}^{2+}$  concentrations, use of gypsum as a Ca source may help ameliorate this problem (Zoca and Penn, 2017; Caires and Guimarães, 2018).

Considering that most eucalyptus forests in Brazil are planted on low fertility soils (Leite et al., 2011), and that long intervals of no-tillage combined with litter turnover lead to soil chemical stratification (Rocha et al., 2018; Tiecher et al., 2018), we hypothesized that gypsum application would promote higher nutrient absorption by eucalyptus trees. Therefore, our objective was to evaluate levels of macronutrients, micronutrients, and potentially toxic elements in eucalyptus leaves following gypsum application.

## 2 MATERIALS AND METHODS

Forests of the hybrid *Eucalyptus urograndis* (*E. urophylla* x *E. grandis*) were cultivated at two locations in the subtropical state of Paraná, Brazil: Jaguariaíva (24°15'04''S, 49°42'21''W, 850m altitude); and Ventania (24°14'45''S, 50°14'34''W, 990m altitude). Climates were Cfb for Jaguariaíva and Cfa for Ventania according to Köppen classification (Alvares et al., 2013).

These areas were located on the second plateau of Paraná. Soils were derived from the Furnas sandstone, Ponta Grossa shale, and Itararé group formed during the Paleozoic era. Soils were classified as Dystrophic Oxisols with a sandy-loam texture. For initial characterization (Table 2), soil samples were collected (0 – 0.10, 0.10 – 0.20, 0.20 – 0.40 m depths), air dried, and sieved (< 2 mm). Analyses of pH,  $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  (KCl 1 mol L<sup>-1</sup> extraction),  $\text{K}^{+}$ , and P (Mehlich I extraction) were conducted according to methodology described by Embrapa (1997).

At each location, experiments utilized a randomized block design. Gypsum (19% Ca and 16% S) was applied to 28 plots, corresponding to six rates (0.3, 0.6, 1.2, 2.4, 4.8, and, 9.6 t ha<sup>-1</sup>) plus control with four replications. Each plot measured 24 x 24 m (576 m<sup>2</sup>) for a total experimental area (per location) of 16,128 m<sup>2</sup>. Experimental areas were previously cultivated with *Pinus* spp for 15 years; 2 Mg ha<sup>-1</sup> of dolomitic limestone (effective calcium carbonate equivalent = 82%) was broadcast onto the soil surface prior to study initiation. In addition,

200 kg ha<sup>-1</sup> of reactive natural phosphate (29% P<sub>2</sub>O<sub>5</sub>) was applied during subsoiling (45 cm depth).

*E. urograndis* seedlings were transplanted on 3 m x 3 m spacings between plants and between rows for a total of 64 plants per plot. At transplanting, N (8.5 kg ha<sup>-1</sup>), P<sub>2</sub>O<sub>5</sub> (51 kg ha<sup>-1</sup>) and K<sub>2</sub>O (17 kg ha<sup>-1</sup>) were applied. Three months later, additional fertilizer was applied at rates of 24 kg N ha<sup>-1</sup>, 8 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, 48 kg K<sub>2</sub>O ha<sup>-1</sup>, and 0.8 kg B ha<sup>-1</sup>. Side dressing sources were urea (N), triple super phosphate (P<sub>2</sub>O<sub>5</sub>), potassium chloride (K<sub>2</sub>O), and boric acid (B).

At 12, 24, and 36 months after transplanting, foliar samples were collected from fresh leaves of four branches at each cardinal point. All samples were dried in a continuous air flow oven at 60°C and milled (< 0.8 mm). Samples were subjected to microwave assisted digestion using HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> as extractors (Embrapa, 2009). Aluminum, B, Ca, Cu, Fe, K, Mg, Mn, P, S, and Zn were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES).

Using the statistical software ASSISTAT 8, results were subjected to variance analysis (ANOVA) and their means were compared via regression analysis (Silva and Azevedo, 2002).

### 3 RESULTS AND DISCUSSION

Although experimental sites had the same soil class classification and very similar initial chemical status (Table 1), macronutrient and Al concentrations in leaf tissue from Jaguariaíva were greater than those from Ventania (Figure 1). Small differences in microclimate and soil nutrient availability probably lead to differential use of soil amendments.

Table 1. Chemical characterization of soils before the experiment initiation at two study areas (municipalities of Jaguariaíva and Ventania – Paraná state, Brazil).

Depth	pH <sup>1</sup> CaCl <sub>2</sub>	pH <sup>2</sup> SMP	Al <sup>3+</sup>	H+Al	Ca <sup>2+</sup>	Mg <sup>2+</sup>	K <sup>+</sup>	BS <sup>3</sup>	CEC <sup>4</sup> pH 7,0	P	C <sub>org</sub>	V <sup>5</sup>	m <sup>6</sup>
m			----- cmol <sub>c</sub> dm <sup>-3</sup> -----						mg dm <sup>-3</sup>	g dm <sup>-3</sup>	----- ---	%	---
Jaguariaíva													
0.00 – 0.20	3.9	5.7	1.5	6.4	0.2	0.2	0.02	0.42	6.82	1.0	23.6	6.1	78.1
0.20 – 0.40	3.8	5.9	1.2	2.2	0.1	0.1	0.02	0.22	2.42	0.7	16.1	9.1	84.5
0.40 – 0.60	4.0	6.1	1.0	4.6	0.1	0.1	0.01	0.21	4.81	0.6	17.4	4.4	82.6

Ventania													
0.00	–												
0.20	4.3	6.2	1.5	4.3	0.1	0.1	0.03	0.23	4.53	2.2	21.0	5.1	86.7
0.20	–												
0.40	4.5	6.7	1.1	3.0	0.1	0.1	0.03	0.23	3.23	1.6	11.0	7.1	82.7
0.40	–												
0.60	4.4	6.8	1.2	2.7	0.1	0.1	0.03	0.23	2.93	0.4	7.3	7.8	83.9

<sup>1</sup>pH CaCl<sub>2</sub> = soil/CaCl<sub>2</sub> 0.01 M ratio 1: 2.5; <sup>2</sup>pH<sub>SMP</sub> = SMP buffer solution; <sup>3</sup>BS = base sum (Ca+Mg+K); <sup>4</sup>CEC = Cation Exchange Capacity at pH 7,0; <sup>5</sup>V = base saturation of CEC (Cation Exchange Capacity); <sup>6</sup>m = aluminum saturation; \*H + Al extraction acetate Ca (0.5 mol L<sup>-1</sup>); Al, Ca and Mg extraction KCl (1 mol L<sup>-1</sup>); K and P extraction Mehlich I; C – organic carbon (volumetric method potassium dichromate).

Leaf Ca concentration were generally higher in the first two years and decreased in the third year (Figure 1) at both sites. The same pattern was observed for K at Ventania. In contrast, higher values of Mg, P, and S (Figure 1) were observed in the third year compared to years one and two. Our results were similar to those reported in the literature (Table 2). Changes in elemental concentrations may be related to exploration of different soil volumes and layers as well as changes in nutrient cycling processes. The use of low release P (natural reactive phosphate) may explain increases of this nutrient observed in the third year. Since our study indicated that foliar nutritional status was influenced by tree age, multiple years of observations may be needed to understand nutrient requirements and dynamics in these forest systems.

Compared to findings by others, Ca values were higher, K and P were lower, and average Mg and S values were within the range defined by previous research (Figure 1 and Table 2). The lower concentrations of K and P were unexpected since both nutrients were supplied by fertilization.

Table 2. Macronutrient concentrations (g kg<sup>-1</sup>) in *Eucalyptus* spp. leaf tissue at different development times in Brazil studies.

		Concentration of macronutrients (g kg <sup>-1</sup> ) in leaves				
Specie	Age	Phosphorus	Potassium	Calcium	Magnesium	Sulfur
<i>E. urograndis</i> <sup>5</sup>	2 months	2.7 – 3.5	14.5 – 21.0	10.0 – 15.9	2.4 – 3.0	1.8 – 3.0
<i>E. urophylla</i> x <i>E. grandis</i> <sup>3</sup>	3 months	1.5 – 2.5	7.9 – 23.8	10.6 – 19.4	2.4 – 3.0	0.6 – 0.8
<i>E. urophylla</i> x <i>E. grandis</i> <sup>4</sup>	5 months	2.1 – 2.7	10.0 – 14.8	8.3 – 13.3	1.9 – 2.2	----
<i>E. grandis</i> <sup>1</sup>	18 months	1.3 – 1.9	3.4 – 4.9	3.6 – 5.2	1.8 – 2.5	1.0 – 1.2
<i>E. saligna</i> <sup>2</sup>	20 months	0.9 – 1.4	----	4.3 – 20.2	1.5 – 3.0	----

<i>E.microcorys</i> <sup>7</sup>	24 months	1.2 – 1.9	7.1 – 11.0	5.4 – 9.4	1.5 – 2.7	----
<i>E. urophylla</i> <sup>6</sup>	36 months	0.9 - 1.4	5.4 – 10.0	2.7 – 11.2	2.9 – 4.1	1.1 – 2.0

Source: Silva et al. (2008)<sup>1</sup>; Guedes and Poggiani (2003)<sup>2</sup>; Medeiro et al. (2016)<sup>3</sup>; Santos et al. (2007)<sup>4</sup>; Rocha et al. (2013)<sup>5</sup>; Assis et al. (2006)<sup>6</sup>; Fortes et al. (2016)<sup>7</sup>.

Considering treatments effects over time at the Jaguariaíva site, macronutrient concentrations decreased in the following order: Ca > K > Mg > S > P. At the Ventania site, the order was Ca > Mg ≈ K > S > P. These sequences basically followed the nutrient availability of the applied soil amendments. Evaluating irrigation effects on nutrient concentration of *Eucalyptus grandis* seedlings in São Paulo state (Brazil), Lopes et al. (2007) found that seedlings accumulated K > Mg > Ca > S > P under 6 mm day<sup>-1</sup>; under 14 mm day<sup>-1</sup>, the accumulation was Ca > Mg > K > P > S. This indicates that, in addition to soil nutrient availability, water availability plays an important role in nutrient uptake and translocation. At the Ventania site, mean annual rainfall (2,043 mm year<sup>-1</sup>) was slightly lower than at Jaguariaíva (2,965 mm year<sup>-1</sup>), which may have contributed to variation in the nutrient accumulation sequence.

The effect of gypsum on Ca tissue levels varied by site and tree age. At Ventania, there was no effect of gypsum over the entire study period (Figure 1 D). In contrast, leaves at the Jaguariaíva site displayed a 2-fold increase ( $p < 0.05$ ) in Ca concentration in the first year (Figure 1 C). In the second year, the highest gypsum rate promoted a Ca leaf concentration that was 55% higher than the control ( $p < 0.01$ ). No treatment effect was observed in the last year. Although the highest rates promoted the highest accumulations, these accumulations dropped about 5 times by the third year when viewing treatment effects over time. In the control treatment, accumulation was only reduced by 3 times between the first and third year. Since Ca is required for trunk and bark formation, this decrease in leaf Ca was expected (Laclau et al. 2000; Hawkesford et al. 2012).

Since applied gypsum contained 16% S, greater treatment effects on leaf S accumulation were expected. By 12 months, Jaguariaíva leaves from the 9.6 T ha<sup>-1</sup> gypsum treatment only accumulated 40% more S than the control ( $p < 0.01$ ; Figure 1 L). However, no treatment effect was observed in subsequent years with rates displaying similar trends over time. For all treatments, the level of S doubled between the first and last experimental year. Results were slightly more significant for Ventania (Figure 1 M), where the two highest rates promoted higher S levels in all years ( $p < 0.05$ ).



Evaluating the influence of gypsum ( $1.2 \text{ ton ha}^{-1}$ ) on eucalyptus for 30 months, Macana (2017) found increased foliar concentrations up to 18 and 6 months following application for Ca and S, respectively. In addition, Rodrigues et al. (2016) reported an influence of gypsum application on Ca and S leaf levels at 18 months. These results agree with the general statement that gypsum enhances Ca and S in foliar tissue for the first and second years after application for different soils, climates, and plants species (Shainberg et al. 1989).

The increase in Mg over time was expected due to its enzymatic role and structural functions in chlorophyll (Laclau et al. 2000; Hawkesford et al. 2012). While a treatment effect ( $p < 0.01$ ) was observed in the two first years at Jaguariaíva (Figure 1 G), the effect at Ventania was only observed at 36 months ( $p < 0.05$ ), primarily due to the sharp drop observed at the highest rate (Figure 1 H). Gypsum has been reported to affect Mg concentrations in soil due to replacement by Ca allowing Mg to move into interstitial soil water (Kost et al. 2018). However, our results showed no strong treatment effect on Mg accumulation in eucalyptus leaves. For K, another element that is supposed to be affected by gypsum application, treatments only differed in the first year ( $p < 0.05$ ) at Jaguariaíva (Figure 1 E, F).



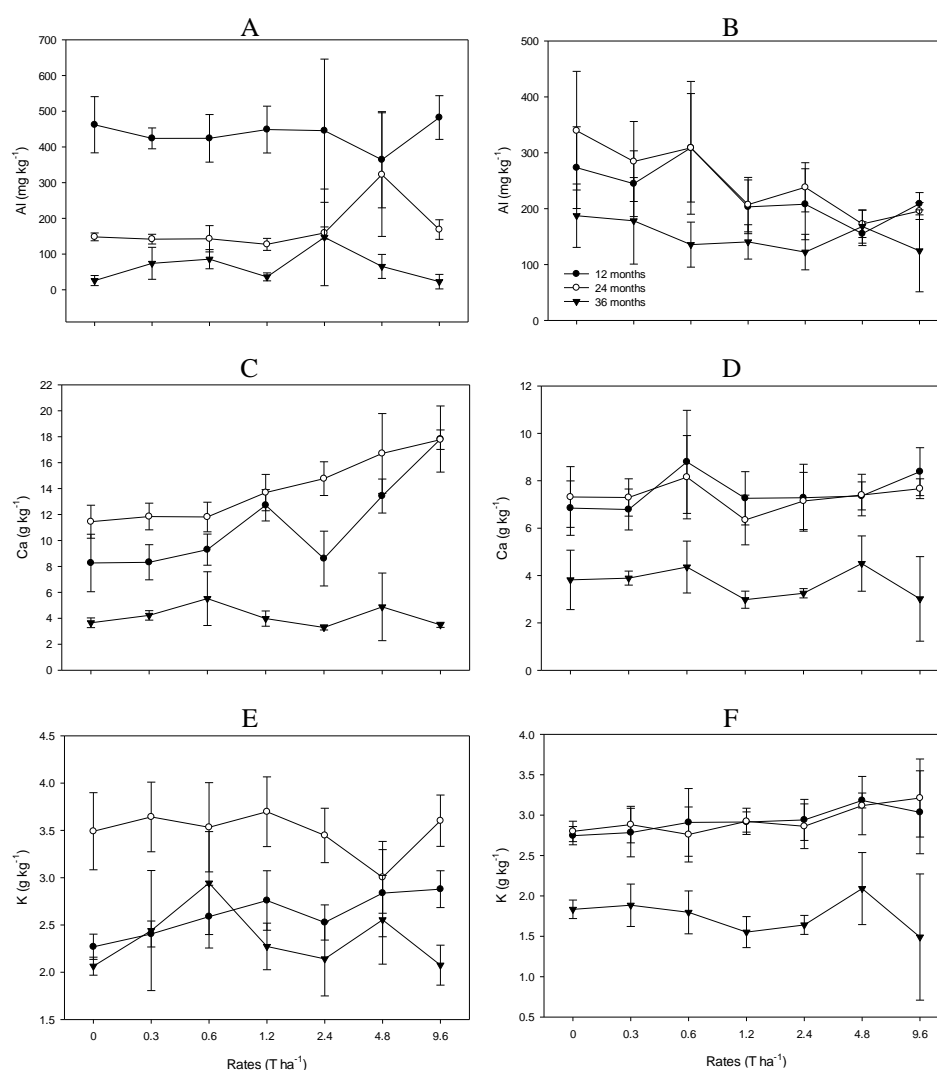


Figure 1. Levels of macronutrients and Al in leaves of *Eucalyptus urograndis* (12, 24, and 36 months after transplanting) cultivated under seven rates of gypsum (0; 0.3; 0.6; 1.2; 2.4; 4.8; and 9.6 T ha<sup>-1</sup>) in two subtropical sites of Brazil: Jaguaraiá (A; C; E; G; I; and L) and Ventania (B; D; F; H; J; and M).

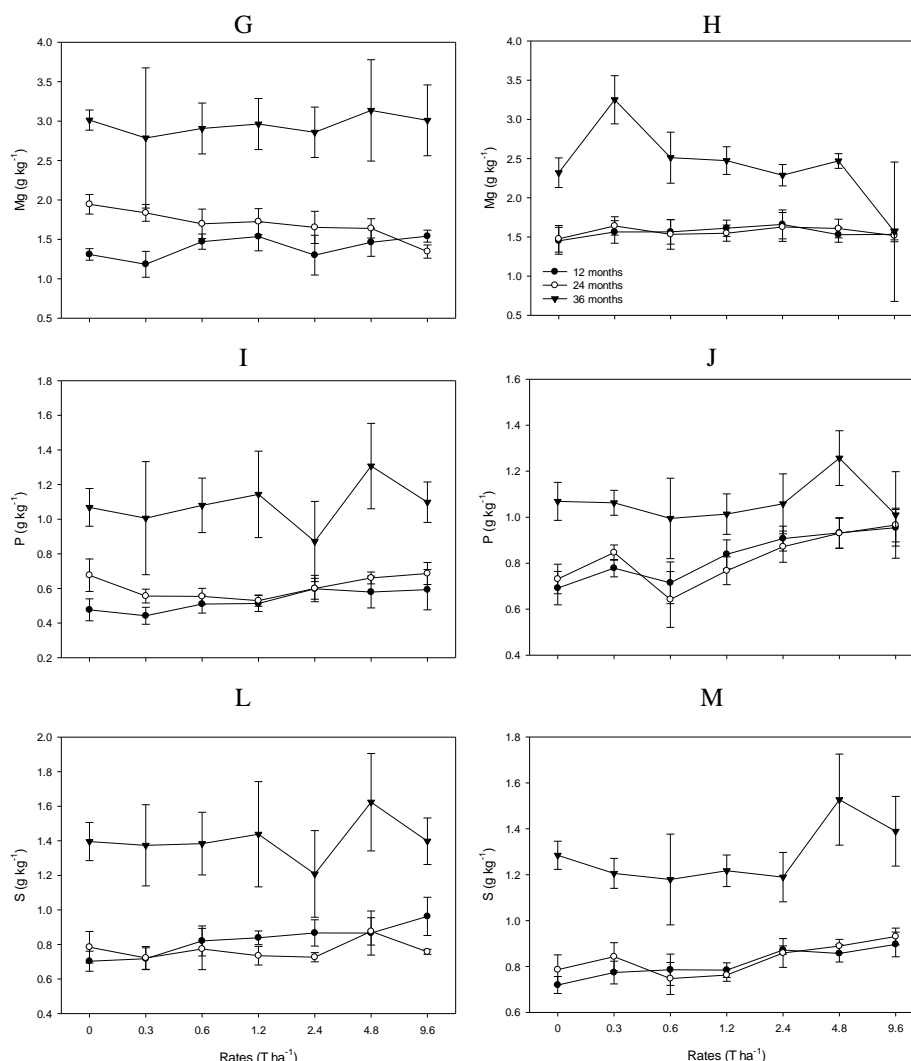


Figure 1 (...Continued). Levels of macronutrients and Al in leaves of *Eucalyptus urograndis* (12, 24, and 36 months after transplanting) cultivated under seven rates of gypsum (0; 0.3; 0.6; 1.2; 2.4; 4.8; and 9.6 T ha<sup>-1</sup>) in two subtropical sites of Brazil: Jaguariáiva (A; C; E; G; I; and L) and Ventania (B; D; F; H; J; and M).

Due to high dissolution product mobility, gypsum is often very effective in alleviating subsoil acidity issues by shifting soluble Al to less toxic forms and decreasing Al activity (Zoca and Penn 2017). However, gypsum application had little or no effect on Al levels in leaf tissues at Jaguariáiva (Figure 1 A). At Ventania, leaf samples displayed a trend for decreased Al ( $p < 0.05$ ), but these values stabilized by the third year (Figure 1 B).

Zambrosi et al. (2007) pointed out that in systems with little or no tillage, due to higher organic matter content, the organic fraction of the soil has a greater capacity to complex cations, especially Al. For this reason, the effectivity of gypsum to neutralize Al is reduced in these soils. The soil at Ventania site had lower surface and subsurface levels of organic matter, and gypsum application promoted lower Al levels in foliar tissue. Regardless of soil

mechanisms, Rocha et al. (2019) noted that eucalyptus trees displayed low accumulation in leaves due to  $Al^{3+}$  resistance.

Gypsum application can stimulate root development (Shainberg et al. 1989; Zoca and Penn 2017), which increases P uptake. In addition, eucalyptus trees are highly efficient in P absorption and retranslocation (Foltran et al. 2019). In our study, gypsum application had little impact on P accumulation during the first two years and none in the last year (Figure 1 I, J). However, a clear trend for P accumulation as a function of higher gypsum rates was seen in Ventania, with a steady increase up to 24 months.

When evaluating effects of gypsum application on micronutrient concentrations (Figure 2), the lack of significant variation among samples in most treatments was associated with high standard deviations. Kost et al. (2018) also reported little effect of gypsum on B and Fe in crop plant tissues. Shainberg et al. (1989) found that Mn uptake was increased following gypsum application. In our study, a very slight increase in Mn was seen at Jaguariaíva (Figure 2 G), while the Ventania site showed a decreasing trend (Figure 2 H). These finding may be due to a dilution effect (B, Fe and Mn) associated with the large increase in leaf area during 36th month experimental period (Melo et al. 2016).

There was no statistical difference for As, Ba, Cd, Cr and Pb related to applied gypsum rate; thus, data were presented in boxplot format (Figure 3). Average levels of these elements were somewhat higher at Jaguariaíva than Ventania. The decreasing trend over time suggests that these elements were primarily absorbed in the first two years of tree growth.

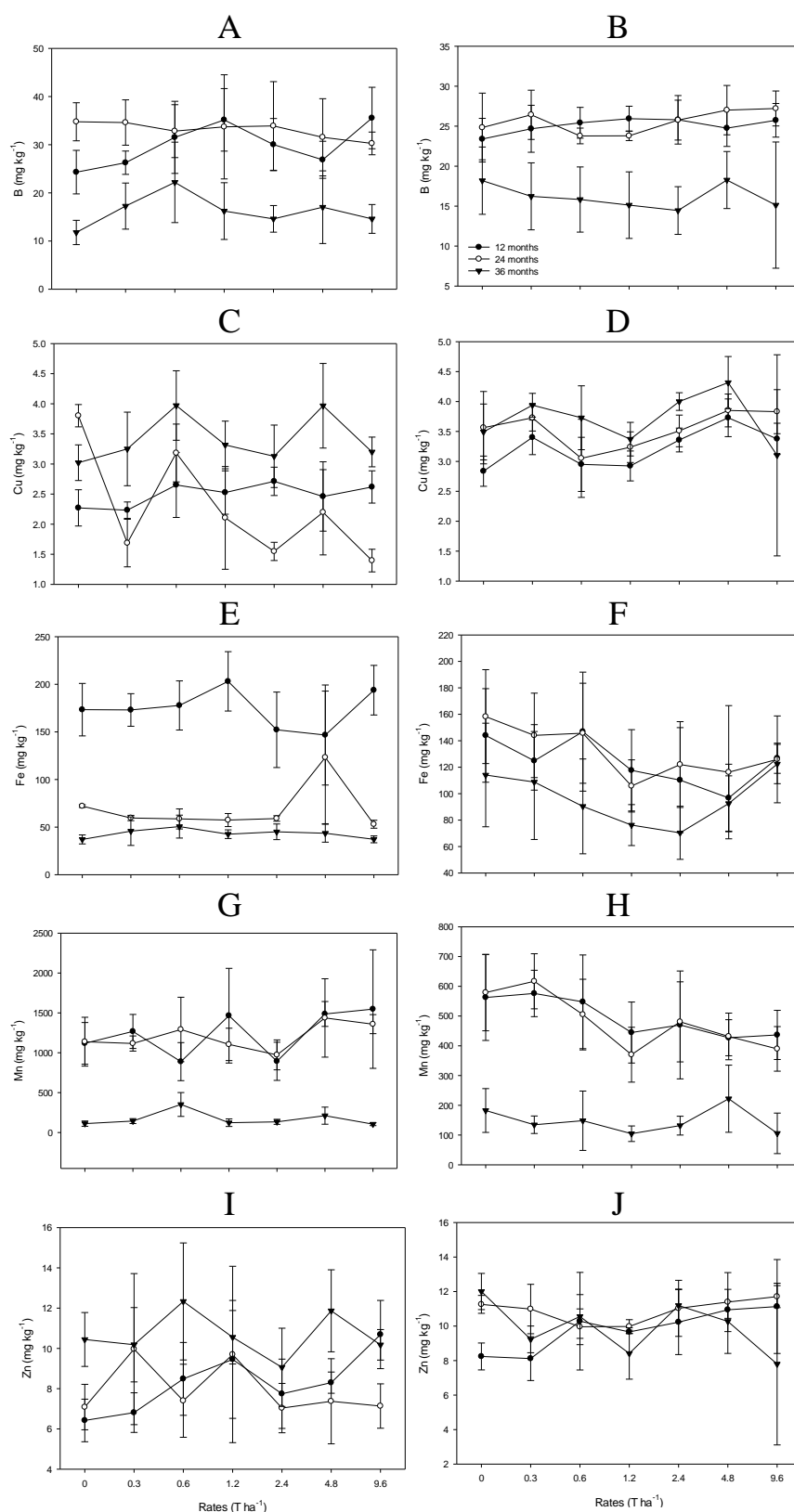


Figure 2. Levels of micronutrients in leaves of *Eucalyptus urograndis* (12, 24, and 36 months after transplanting) cultivated under seven rates of gypsum (0; 0.3; 0.6; 1.2; 2.4; 4.8; and 9.6 T ha<sup>-1</sup>) in two subtropical sites of Brazil: Jaguariáiva (A; C; E; G; and I) and Ventania (B; D; F; H; and J).

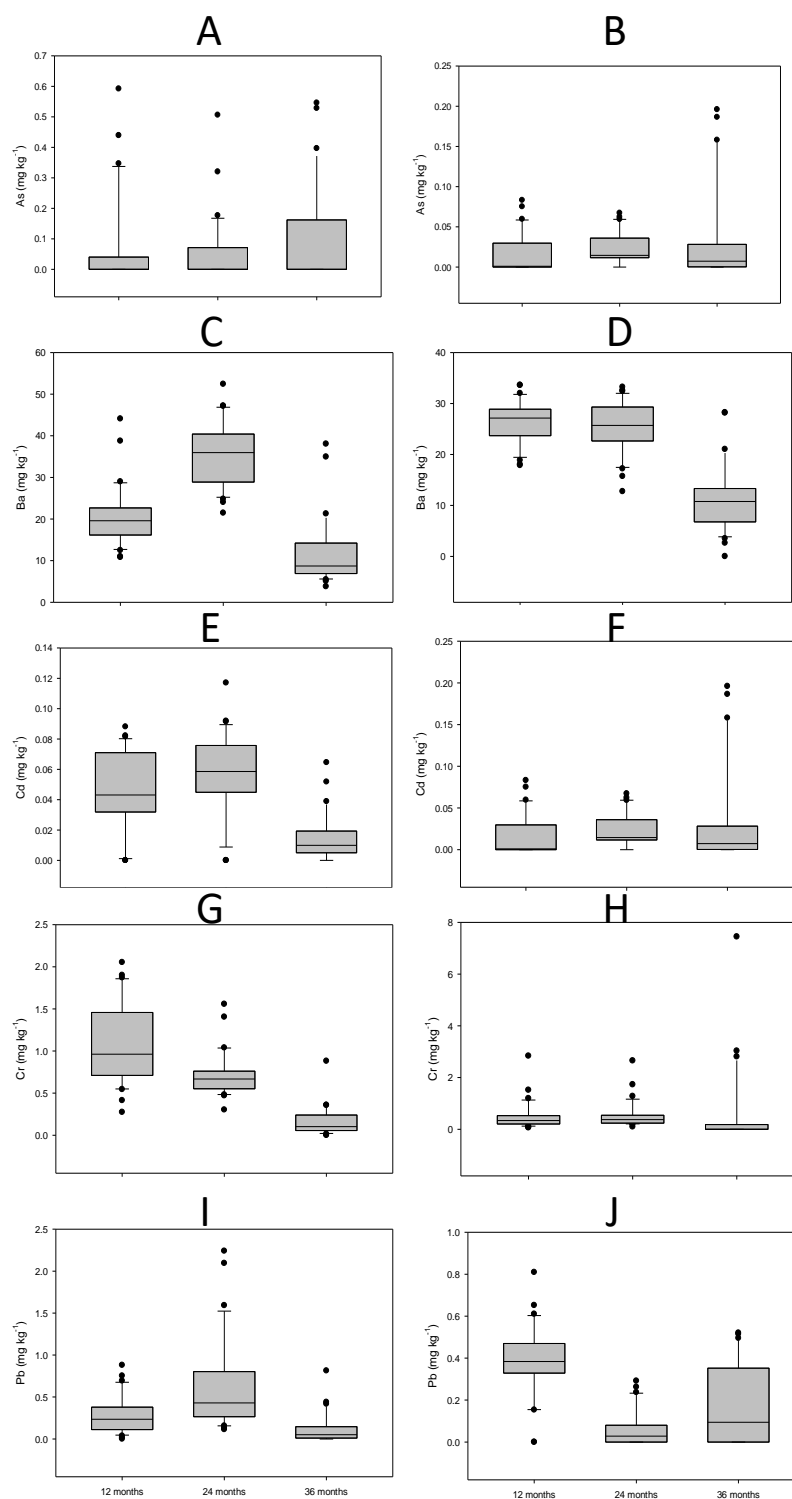


Figure 3. Levels of potentially toxic elements in leaves of *Eucalyptus urograndis* (12, 24, and 36 months after transplanting) cultivated under seven rates of gypsum (0; 0.3; 0.6; 1.2; 2.4; 4.8; and 9.6 T ha<sup>-1</sup>) in two subtropical sites of Brazil: Jaguariáiva (A; C; E; G; and I) and Ventania (B; D; F; H; and J).

Since foliage is the largest above-ground sink of nutrients, consideration of leafy crown development is essential for understanding overall tree nutrient dynamics (Miller 1995). Our data showed that gypsum application had little or no effect on the accumulation of macronutrients, micronutrients, and potentially toxic elements in *E. urograndis* under subtropical conditions. When present, effects were more evident in the first two years of growth. Tiecher et al. (2018) recommended gypsum application to tropical soils when Al saturation was above 20% and/or exchangeable Ca content was below  $0.5 \text{ cmol}_c \text{ dm}^{-3}$  in the 0.20-0.40 m soil layer. Although the soils at Jaguariaíva and Ventania met these criteria, the low effects of gypsum application were probably due to a good nutrient balance obtained by fertilizer and lime applications (Zoca and Penn 2017); under this scenario, gypsum played a secondary role in tree nutrition. Kost et al. (2018) also observed no consistent yield responses to gypsum materials, but correlation values tended to be greater for gypsum application rates higher than  $9 \text{ T ha}^{-1}$ .

Eucalyptus development can be divided into three phases: before, during, and after canopy closure. When water and nutrient supplies are adequate during the period before closure, maximal photosynthetic activity is associated with rapid development of the crown and root system. During this phase, responses to fertilization are very common. After canopy closure when the availability of light and water becomes more limiting, the establishment of nutrient cycling reduces or neutralizes responses to fertilization. At this stage, nutrient demands are met by litter mineralization (biogeochemical cycling) and internal nutrient translocation (biochemical cycling) (Miller 1995; Gonçalves 2010). According to Da Silva et al. (2012), the order of nutrient accumulation in litter is  $\text{N} > \text{Ca} > \text{S} > \text{Mg} > \text{K} > \text{P}$ . This may help explain the lack of treatment effects in our study.

Most published literature has focused on the impacts of gypsum/phosphogypsum on soil properties rather than crop yields (Zoca and Penn 2017). Tiecher et al. (2018) observed that crops can respond positively to gypsum in the simultaneous presence of high soil subsurface acidity and water deficiency. When gypsum was applied to soils with low subsurface acidity (Al saturation  $< 10\%$ ), adequate exchangeable Ca content ( $> 3.0 \text{ cmol}_c \text{ dm}^{-3}$ ), and available P and S, crop yield failed to increase. The effect of time is also important in the movement of elements within the soil profile (Zoca and Penn 2017), thus long-term impacts of gypsum applications may not be adequately reflected in short-term (i.e., 2-3 years) studies (Kost et al. 2018).

**4 CONCLUSION**

Despite similar soil properties at the two experimental sites, the wide variation of nutrients and Al concentrations observed in foliar tissue suggest the influence of other factors such as weather conditions. Tree age also influenced foliar concentrations with general trends of diminishing Al, Ca, K, B, Fe, and Mn and increasing Mg, P, and S. Root system dynamics and nutrient cycling processes could be influencing these trends, which suggests that tree age is an important factor when comparing eucalyptus nutritional status among experiments. Despite the wide range of gypsum rates, nutritional effects and site responses were variable. Higher gypsum rates enhanced foliar Ca concentration at Jaguariaíva in the first two years but had no effect in the third year. Gypsum increased S concentration in the first year at Jaguariaíva for the highest rate and in all three years at Ventania for the two highest rates. The application of gypsum as a Ca source seemed to decrease Mg tissue concentration for both sites but at different time scales. Diminishing Al and Mn and increasing P in foliage was observed at Ventania. The influence of sites and time seems to have had more impact than gypsum application. Overall, applying gypsum to eucalyptus in subtropical Brazil had little effect on foliar accumulation of micronutrients, macronutrients, and potentially toxic elements.

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