

FOLIAR COMPOSITION OF *Eucalyptus urograndis* IN TWO SITES FERTILIZED WITH N, P AND K IN SOUTHERN BRAZIL.

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Resumo

Composição foliar de Eucalyptus urograndis em dois sítios adubados com N, P e K no sul do Brasil. A composição foliar vem sendo utilizada como ferramenta no estabelecimento da diagnose nutricional e avaliação da efetividade do uso de nutrientes. Estes conhecimentos são ainda escassos sob condições subtropicais de cultivo. Neste sentido, o estudo tem por objetivo avaliar o efeito da fertilização com N, P e K na concentração foliar de *Eucalyptus urograndis*. Os experimentos foram instalados em Latossolo Vermelho-Amarelo Distrófico textura franco-arenosa, no município de Jaguariaíva e Ventania, estado do Paraná. No experimento com N e P foram testadas as seguintes doses: 0; 30; 60; 120 e 240 kg ha⁻¹ de N e P₂O₅; e 0; 35; 70; 140 e 280 kg ha⁻¹ de K₂O, parcelado em quatro vezes, uma no plantio e três de cobertura aos 3, 9 e 12 meses de idade. A resposta foi avaliada em função do estado nutricional das plantas aos 12, 24 e 36 meses, via concentração foliar. Também avaliamos os níveis de deficiência de potássio, através da análise visual e química. A aplicação do N e P não resultou em alteração nos teores foliares dos mesmos em todos os períodos avaliados e nas duas regiões, ocorrendo uma leve diminuição das concentrações com o tempo. Efeito diferente ocorreu com o K no sítio de Jaguariaíva que alterou sua concentração foliar no 24º e 36º mês, e ocorreu aumento das concentrações foliares com o tempo. Esses efeitos demonstram que muitas vezes o diagnóstico nutricional, com base nas concentrações foliares, podem apresentar limitações de interpretação e não deve ser avaliado isoladamente para recomendação de fertilização.

Palavras-chave: nutrição florestal, fertilização florestal, subtropical, eucalipto, faixas críticas.

Abstract

The leaf composition has been used as a tool in establishing the nutritional diagnosis and evaluation of the effectiveness of the use of nutrients. This knowledge is still scarce in subtropical conditions of cultivation. In this sense, the study aims to evaluate the effect of N, P and K fertilization on leaf concentration of *Eucalyptus urograndis*. The experiments were installed in the Oxisols of the sandy-loam texture, in the counties of Jaguariaíva and Ventania, state of Paraná. In the N and P experiments, the following doses: 0; 30; 60; 120 and 240 kg ha⁻¹ of N and P₂O₅; and 0; 35; 70; 140 and 280 kg ha⁻¹ K₂O, split in four applications: during planting and side dressing at 3, 9 and 12 months of age. The response was evaluated according to the nutritional status of the plants at 12, 24 and 36 months, by foliar concentration. It also evaluated levels of K deficiency plant through visual and chemical analysis. The application of N and P did not result in alteration in the leaf contents of the same in all evaluated periods and in the two regions, with a slight decrease in the concentrations with time. A different effect occurred with K at the Jaguariaíva site, which altered its foliar concentration at the 24th and 36th month, and increased foliar concentrations with time. These effects demonstrate that foliar diagnosis, through foliar concentrations, may present limitations of interpretation and should not be evaluated separately for fertilization recommendation.

Keyword: forest nutrition, forest fertilization, subtropical, eucalypt, tracks critical.

INTRODUCTION

After two or three cycles with *Pinus taeda* L. or *Pinus elliottii* Engelm var. *elliottii* under subtropical climate, many of the reforested areas in southern Brazil has been replaced by eucalyptus plantations. Due to the exchange and utilization of fast-growing species, as well as being more nutrient demanding, new plantings are likely to require improved fertility, but information on the need for fertilization and evaluation of eucalyptus nutritional status in this new condition is little known.

Critical nutrient levels have been established in many countries for most species to assist in recommending fertilizers (MILLNER; KEMP, 2012). In Brazil, Silveira *et al.* (2000) and Gonçalves (1995)

established critical leaf ranges for eucalyptus, without species definition, under tropical condition. Both authors established critical values of N very close, between 21 to 30 g kg⁻¹. However, for P and K the ranges are distinct for what may be considered appropriate, Silveira *et al.* (2000) and Gonçalves (1995) established a range of 1.7 to 2.2 and 1.0 to 1.3 g kg⁻¹ P, respectively. Silveira *et al.* (2000) also defined higher and narrower requirements for K being between 8.5 to 9.0 g kg⁻¹ than the range of between 5.5 to 8.5 g kg⁻¹ indicated by Gonçalves (1995). Note that the maximum levels of the second author would be deficient for the first. This is probably due to the selection of the most efficient varieties of eucalyptus for nutrient utilization (FARIA *et al.*, 2008). Regarding Ca this range was also different 7.1 to 11 g kg⁻¹ for Silveira *et al.* (2000) and 3.5 to 6.0 g kg⁻¹ for Gonçalves (1995), while Mg can be considered between 2.0 to 3.0 g kg⁻¹ for both authors.

Regarding micronutrients the values are relatively close, Silveira *et al.* (2000) determined for Cu, Zn, B, Fe and Mn the following values 6 to 7, 15 to 20, 34 to 44, 65 to 125 and 200 to 840 mg kg⁻¹, respectively. Gonçalves (1995) presented 7 to 10, 10 to 18, 30 to 60, 70 to 200 and 100 to 800 mg kg⁻¹ for Cu, Zn, B, Fe and Mn, respectively.

Important information that does not appear in the nutritional diagnosis manuals is the age of reforestation, responsible for large changes in concentrations over time, site conditions (Gonçalves *et al.*, 2004) and also due to the clonal variety of the hybrid. Laclau *et al.* (2000) found variations in leaf tissue from the first to the seventh year for *Eucalyptus urograndis* in Congo, showing an upward trend for N, a decrease for P, K, Ca and Mg. In tropical conditions in Brazil and for a wide variety of *E. urograndis* some studies also show this variation in concentration over time and the results vary until the 36th month. Silva *et al.* (2013) found a downward trend for P and K, and an increase for Ca, while N and Mg showed variable behavior, with oscillations over time. For Melo *et al.* (2016) there was increase for N, oscillation for P and decrease for K.

Thus, the great divergence regarding the leaf concentration behavior is noted. In Brazil, the critical ranges for eucalyptus are outdated, containing little information by species, tree age and mainly for subtropical conditions. So, this work aims to obtain information regarding the nutritional status of clone *Eucalyptus urograndis* under doses of N, P and K applied separately in two traditional forest regions in southern Brazil. It was hypothesized that the critical ranges used in Brazil could be not to sites located in subtropical condition and forest soils.

MATERIAL AND METHODS

Study area characterization

The experimental units were installed in the municipalities of Jaguariaíva and Ventania, both in the state of Paraná, at the geographic coordinates 24° 15'12"S and 49° 40' 40" O and altitude of 926 m for the first, and 24° 01'17,25"S and 50° 15' 39.62" O and altitude of 718 m for the second. We implemented the experiment in December 2013 for Jaguariaíva and April 2014 for Ventania. In both municipalities, the soil is classified as Dystrophic Red-Yellow Latosols with sandy-loam texture.

The two study regions are characterized according to Alvares *et al.* (2013), as humid subtropical climate, climate zone C, where Jaguariaíva is in the Cfb type, where summer temperatures are milder, while Ventania is in a transition region between Cfb and Cfa, which is characterized by warmer summers.

The two experimental areas were approximately three hectares each, with pine as the predecessor crop, planted at 3 x 2 m spacing. The plantings were clearcutting by harvester machine. The residues were managed in different ways in each site. Large piece of branch (more than five centimeters in diameter) were collected and took out to use as biomass at the Jaguariaíva site and raked and piled up at the Ventania site.

After harvesting, soil samples were collected to a depth of 100 cm for chemical characterization of the soil (TABLE 1) following the methodology described by EMBRAPA (2009).

Table 1. Chemical characteristics of sites before *Eucalyptus urograndis* planting in Jaguariaíva and Ventania counties, Paraná State – Brazil.

Tabela 1. Características químicas das áreas antes do plantio de *Eucalyptus urograndis* nos experimentos, em Jaguariaíva e Ventania, Paraná, Brasil.

Depth (cm)	pH		Al	H+Al	Ca	Mg	K	SB ¹	CTC ²	P	C	³ V	⁴ m
	CaCl ₂	SMP											
Jaguariaíva													
0-20	3.8	5.7	1.5	6.4	0.2	0.2	0.02	0.4	6.8	1.0	23.6	5.3	81.3
20-40	3.9	5.9	1.2	2.2	0.1	0.1	0.02	0.2	5.5	0.7	16.1	4.0	85.0
40-60	4.0	6.1	1.0	4.6	0.1	0.1	0.01	0.2	6.0	0.6	17.4	4.3	82.7
60-80	4.0	6.2	0.9	4.3	0.1	0.1	0.01	0.2	4.5	0.8	14.0	4.7	80.3
80-100	4.1	6.3	0.8	3.9	0.1	0.1	0.01	0.2	4.1	0.7	10.2	5.0	78.3
Ventania													
0-20	4.3	6.2	1.5	4.3	0.1	0.1	0.03	0.3	4.6	2.2	21.0	5.9	84.6

20-40	4.5	6.7	1.1	3.0	0.1	0.1	0.03	0.2	3.3	1.6	11.0	7.1	82.9
40-60	4.4	6.8	1.2	2.7	0.1	0.1	0.03	0.2	3.0	0.4	7.3	7.8	83.8
60-80	4.5	6.8	1.3	2.6	0.1	0.1	0.02	0.2	2.9	0.8	6.0	7.8	85.4
80-100	4.5	6.9	1.1	2.6	0.1	0.1	0.05	0.3	2.9	2.8	6.4	9.7	78.0

¹SB=Soma of bases; ²Cation Exchange Capacity (CTC=CTC pH=7); ³V=Base saturation; ⁴m=Aluminum saturation.

Installation of sample units

Before planting the seedlings, subsoiling was performed at a depth of 45 cm and 200 kg ha⁻¹ of reactive phosphate rock- RPR (29% of total P₂O₅) was applied at a depth of 25 cm. After subsoiling, 2,000 kg ha⁻¹ of dolomitic limestone (Effective Neutralizing Value or Effective Calcium Carbonate = 82%) was applied to the soil surface without incorporation.

Eucalyptus urophylla x *Eucalyptus grandis* (*Eucalyptus* urograndis, clone AEC 224) seedlings were planted by hand planter over the subsoiling line, spaced 3 x 3 m. After planting, 576 m² plots (8 rows with 8 plants each) were marked to compose the experiments, assembled in random blocks with four replications.

At each site (Jaguariaíva and Ventania) three separate nitrogen, phosphorus and potassium fertilization experiments were conducted. In the experiments to evaluate nitrogen and phosphate fertilization five treatments were installed, corresponding to the rates of 0, 30, 60, 120 and 240 kg ha⁻¹ of N and P₂O₅, respectively. In the experiment to evaluate potassium fertilization, doses of 0, 35, 70, 140 and 280 kg ha⁻¹ of K₂O were used. All doses of all experiments were splitted into four applications (planting, 3, 9 and 12 months).

To complement the fertilization evaluated in the treatments were also applied 8.5, 51 and 17 kg ha⁻¹ of N, P₂O₅, K₂O, respectively. And three other side dressing fertilization were performed at three, nine and 12 months of age with 24, 8, 48 and 0.8 kg ha⁻¹ of N, P₂O₅, K₂O and B, respectively. In each experiment, the element tested was not applied in these complementary fertilizers. The sources used in all fertilizers were urea (46% N), triple superphosphate (45% P₂O₅), potassium chloride (60% K₂O) and boric acid (17% B). Fertilizer application was about 5 cm deep and 15 cm away from the seedling on both sides of the subsoiling line.

Leaf analysis

For leaf analysis, samples were collected from the middle third of the plant of a secondary branch, excluding new and senescent leaves, ie physiologically active leaves, in the four cardinal points of the four central trees of the plot and mixed to take a composite sample.

This procedure was performed in all plots of both experiments at 6, 12, 24 and 36 months at Jaguariaíva site and at 12, 24 and 36 months in Ventania, being that there were not enough leaves at this site for collection and analysis at 6 months. After this stage the leaves were washed in deionized water and completely dried at 65 °C and ground in a Willey mill and submitted to chemical analysis to determine the macronutrients: P, K, Ca, Mg and S, micronutrients: B, Cu, Fe, Mn and Zn, in addition to Al. The analytical methodology was proposed by Martins and Reissmann (2007) which consists of dry digestion at 500 °C with subsequent solubilization in 3 mol L⁻¹ HCl after determination in the plasma emission spectrophotometer. (model: VARIAN 720-ES ICP).

With the processed leaf samples the determination of nitrogen and carbon was performed. For this, this material was again milled in a mechanical mill and weighed 15 mg samples that were packed in tin (Sn) capsules. And then they were running in the elementary analyzer (model: VARIO EL III) to determine the elemental N and C, for the two data were submitted to analysis of variance and when significant effects were detected regression equations, considering the probability of 0.1 %, 1% and 5% ($\rho < 0.01$ and $0.01 < \rho < 0.05$) and the coefficient of determination (r^2) regions in the periods 12, 24 and 36 months.

K deficiency test

Using the experiment with potassium fertilization, a deficiency test was created with the control treatment (0 kg ha⁻¹ K₂O), which was performed to determine the concentration that indicates the potassium deficiency in the leaves. The categories were created through the visual diagnosis of the leaves, considering the most deficient leaf with marginal necrosis from the apex to the base of the leaf, also called inverted “V” symptom. Thus, four categories of disability were created, the first category being severe disability (Level I), moderate disability (Level II), mild disability (Level III) and non-disability (Level IV).

These leaves were analyzed for nutritional content as described in the item leaf analysis. The leaf contents of this test were evaluated by Tukey mean comparison test, and the homogeneity of variances by Barlett test and variance analysis were previously verified.

RESULTS

Macronutrient Concentration

Nitrogen- Nitrogen fertilization did not result in variation in leaf concentration in both sites over 36 months. However, temporal variation was observed with lower values for concentration at 24 months in the Ventania site. In the Jaguariaíva site the values were of greater variability without a clear definition of the time (FIGURE 1 a; b). It is important to consider that in the Ventania site all biomass resulting from the harvest were piled, while in Jaguariaíva all woody material exceeding five centimeters in diameter were removed.

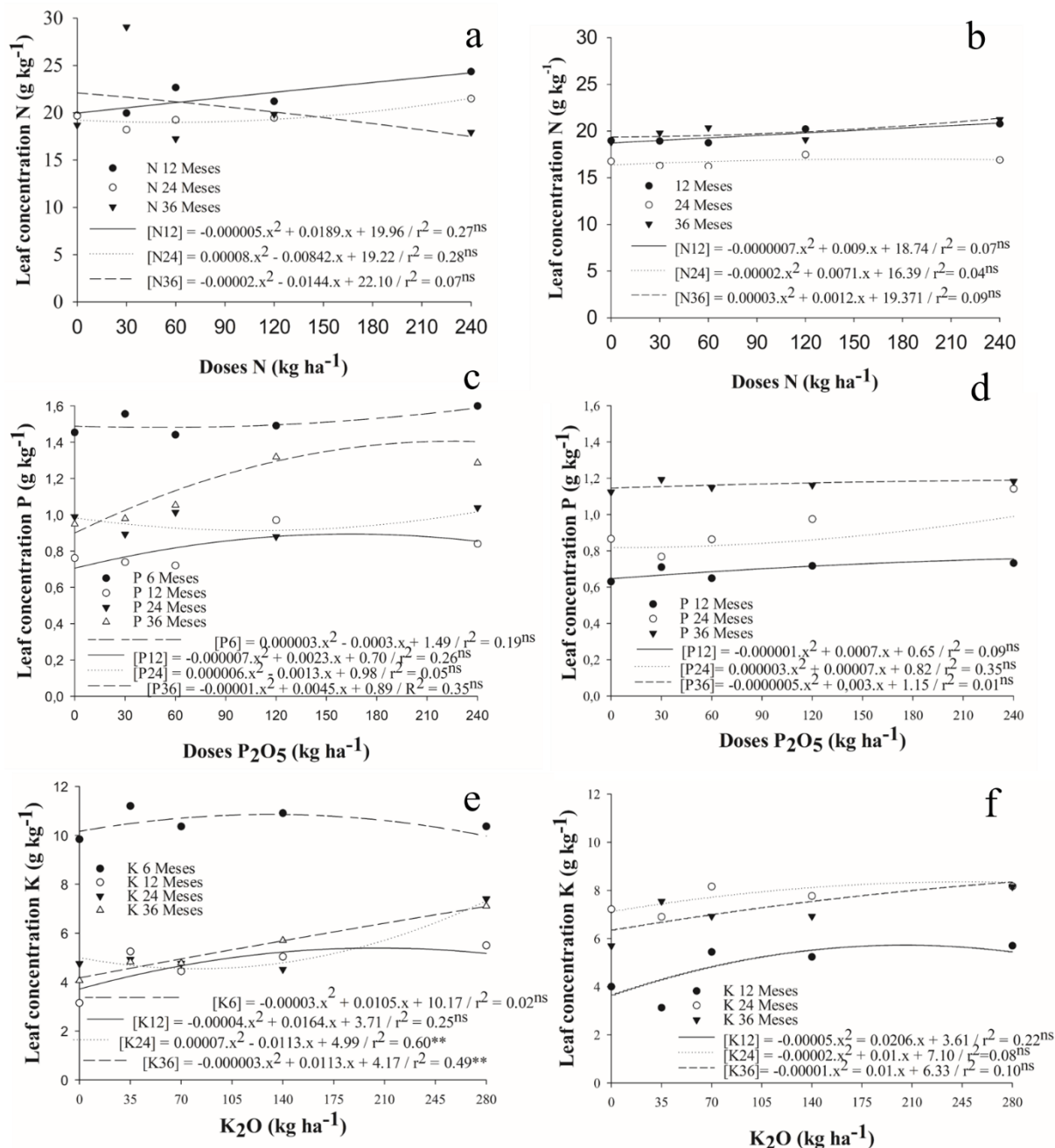


Figure 1. Concentration of nitrogen (a, b), phosphorus (c, d) and potassium (e, f) in leaves depending on the application of 0, 30, 60, 120 and 240 kg ha⁻¹ N and P₂O₅ and 0, 35, 70, 140 and 280 kg ha⁻¹ K₂O in *Eucalyptus urograndis* at the sites of Jaguariaíva (a, c, e) and Ventania (b, d, f), state Paraná, Brazil. ^{ns} not significant, ^{**} significant at 1% probability ($p < 0.01$).

Figura 1. Concentração de nitrogênio (a; b), fósforo (c; d) e potássio (e; f) nas folhas em função da aplicação de 0, 30, 60, 120 e 240 kg ha⁻¹ de N e P₂O₅ e 0, 35, 70, 140 e 280 kg ha⁻¹ K₂O em *Eucalyptus urograndis* nos

sítios de Jaguariaíva (a; c; e) e Ventania (b; d; f), Paraná, Brasil. ^{ns} não significativo, ; ** significativo a 1% de probabilidade ($p < 0,01$).

Phosphorus - The use of P did not increase leaf concentration values during the periods evaluated at both sites (FIGURE 1 c; d). However, it is possible to observe a small tendency of increase at 36 months for leaf concentration in Jaguariaíva (FIGURE 1 c). However, there is a direct relationship between age and P concentration in all treatments, starting at 0.81 g kg^{-1} at 12 months to 1.12 g kg^{-1} at 36 months at the Jaguariaíva site. However, at six months the concentration was 1.51 g kg^{-1} , the highest concentration period. In the Ventania site, a similar trend was observed with an increase from 0.68 g kg^{-1} at 12 months to 1.16 g kg^{-1} at 36 months. Higher concentrations of P in Jaguariaíva site in relation to Ventania site were observed, in the first one the concentrations were $1.51; 0.81; 0.96$ and 1.12 g kg^{-1} at 6, 12, 24 and 36 months, respectively, while in the second it was $0.68; 0.92$ and 1.16 g kg^{-1} at 12, 24 and 36 months, respectively.

Potassium - The concentration of K in the plant was only affected by fertilization for the Jaguariaíva site and only at 24 and 36 months with concentrations of 7.41 and 7.11 g kg^{-1} of K, respectively, at a dose of $280 \text{ kg ha}^{-1} \text{ K}_2\text{O}$ (FIGURE 1 e), that is, nutritional changes occurred one year after fertilizer application was completed. In the treatment without K, symptoms of K deficiency were observed in the inner leaves of the crown. Potassium effect for both sites and in most treatments showed similar behavior, increasing from 12th month to 24th month and decreasing at 36th month (FIGURE 01 e; f). This trend was more pronounced in the Ventania site, on average, the values were $4.70; 7.65$ and 7.05 g kg^{-1} at 12, 24, and 36 months, respectively (FIGURE 1f). In the Jaguariaíva site, on average, the values were $10.5; 4.67; 5.28$ and 5.29 g kg^{-1} at 6, 12, 24 and 36 months, respectively (FIGURE 1 e). It can be highlighted that in both sites the control treatment presented the lowest concentration at 12 and 36 months (FIGURE 01 e, f), and the concentration at the Ventania site was slightly higher than the Jaguariaíva site.

Temporal variation of macronutrients, micronutrients and aluminum in leaf tissue over time

There were changes in macronutrient concentrations (Ca, Mg and K) regardless of the applied N, P and K doses, but with different behaviors, with decrease for Ca and Mg and increase for K (FIGURE 2). Another observation that can be made is the separation by nutritional concentration gradient in the different periods evaluated, at 12 and 24 months in both sites, followed the sequence $\text{Ca} > \text{K} > \text{Mg} > \text{P}$, at 36 months there was an increase in K content by changing the sequence to $\text{K} > \text{Ca} > \text{Mg} > \text{P}$ (FIGURE 2).

The carbon content for both sites was lower at 12 months increasing over time, from 475 and 472 g kg^{-1} at 12 months to 499 and 497 g kg^{-1} at 24 months and 501 and 505 g kg^{-1} at 36 months (FIGURE 2 a; b), respectively.

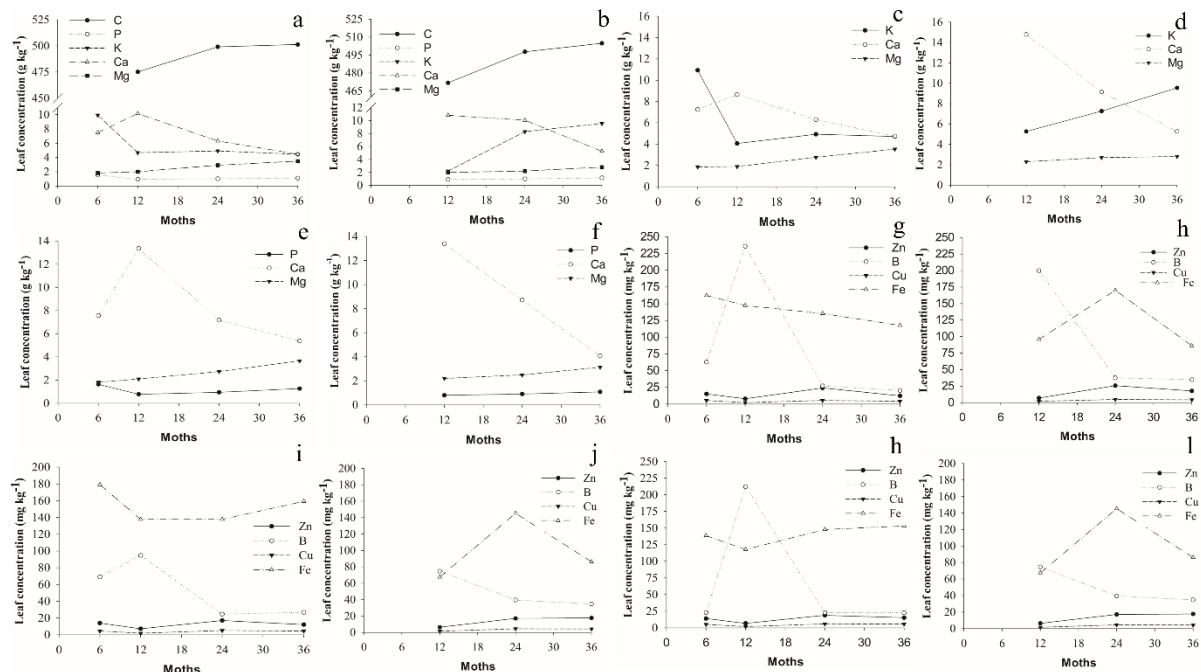


Figure 2. Concentration of nutrients over 36 months of C, P, K, Ca, Mg, Fe, Cu, B and Zn in *Eucalyptus urograndis* for the experiments N (a; b; g; h), P (c; d; i; j), K (e; f; k; l) at the sites of Jaguariaíva (a; c; e; g; i; k) and Ventania (b; d; f; h; j; l), state Paraná, Brazil

Figura 2. Concentração dos nutrientes ao longo de 36 meses de C, P, K, Ca, Mg, Fe, Cu, B e Zn em *Eucalyptus urograndis* para os experimentos N (a; b; g; h), P (c; d; i; j), K (e; f; k; l) nos sítios de Jaguariaíva (a; c; e; g; i; k) e Ventania (b; d; f; h; j; l), PR, Brasil.

Plant age also influenced the concentration of micronutrients, decreasing Fe, B and Cu concentration (FIGURE 2), with its highest concentrations in the first 12 months. The opposite was observed for Zn with increase between 12 and 24 months and stabilizing until the 36th month.

It is also possible to separate the micronutrients by concentration gradient, at 12 months the sequence was B > Fe > Zn > Cu, while at 24 and 36 months the sequence was Fe > Zn ≥ B > Cu.

Elements Al and Mn showed significant reduction in concentration over time (FIGURE 3 a; b). The concentration of Mn at 12 months in all experiments was between 1400 and 1700 mg kg⁻¹, at 24 months the reduction was more pronounced for the Jaguariaíva site, reaching 450 mg kg⁻¹; and then to 300 mg kg⁻¹ at 36 months. While for the Ventania site, the decrease in concentrations from 12 months to 24 months was smaller, and in the end, the concentration was higher than that found in Jaguariaíva. The Al followed the same trend, but with lower values with initial concentrations between 300 to 500 mg kg⁻¹ and at 36 months with concentration closer to 200 mg kg⁻¹ (FIGURE 3 a; b).

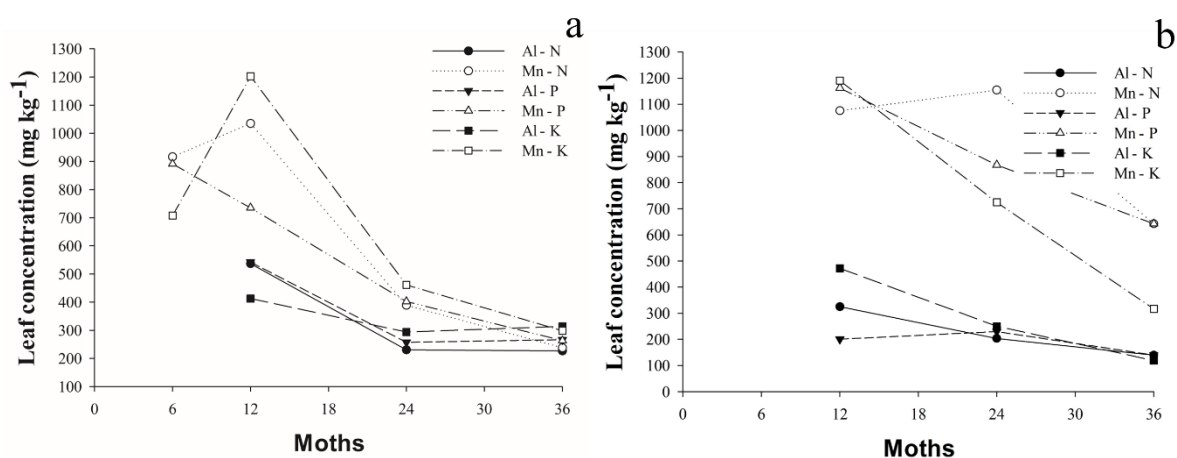


Figure 3. Concentrations of Mn and Al over 36 months in *Eucalyptus urograndis* for N, P and K experiments at Jaguariaíva (a) and Ventania (b), state Paraná, Brazil.

Figura 3. Concentrações de Mn e Al ao longo de 36 meses em *Eucalyptus urograndis* para os experimentos N, P e K em Jaguariaíva (a) e Ventania (b), PR, Brasil.

Potassium Deficiency Test

The K concentrations of leaves with and without symptoms of deficiency showed no statistical difference, although the signs of leaf symptoms are quite evident. The values found were 3.29; 3.33; 3.66 and 3.13 g kg⁻¹ of K for categories I, II, III and IV, respectively (FIGURE 4).



Figure 4. Levels of potassium deficiency in *Eucalyptus urograndis* (clone AEC-224) and foliar concentration.

Figura 4. Níveis de deficiência por potássio em *Eucalyptus urograndis* (clone AEC – 224) e concentração foliar.

DISCUSSION

Macronutrient Concentration

Nitrogen - The concentration range of N observed in the present study, 18 to 21 g kg⁻¹, is within or close to the considered adequate, 20 to 30 g kg⁻¹, proposed by Silveira *et al.* (2000) and Gonçalves (1995) in both locations studied. It may be considered adequate when compared to the range of 18 to 29 g kg⁻¹ proposed by Dell *et al.* (2002). This maintenance of levels close to adequate indicated a high N availability in the soil even after three years, since there was no effect of N fertilization on leaf concentrations. Also, there was no effect of N fertilization on any of the evaluated growth parameters (height, DBH, commercial volume) (BASSACO *et al.*, 2018).

The absence of changes in leaf N concentration corroborates the indication of good availability by the soil or residue from the previous crop. The high concentration of organic C observed in Jaguariaíva (23.6 g dm⁻³) and Ventania (21.0 g dm⁻³) suggests a high availability of N (PULITO *et al.*, 2015). The absence of change in leaf N concentration was also found by Jesus *et al.* (2012) in *Eucalyptus urophylla* clone at 30 months as a function of fertilization, obtaining an average of 19.9 g kg⁻¹ of N leaf.

Thus, the sites studied indicated high sufficiency in N and are likely to remain in the coming years, due to the increase in biogeochemical cycles and mainly by the internal retranslocation of N, which tends to increase from the third year (BARRETO *et al.*, 2012). This condition is reinforced by the fact that small oscillations in leaf N levels over the years (FIGURE 1), indicate the strong biochemical capacity of the plant to maintain this element. **Phosphorus** - Leaf P concentrations were considered deficient since it was below the 1.0 g kg⁻¹ value established by Gonçalves (1995), at 12 and 24 months in Jaguariaíva and Ventania, and at 36 months reached the appropriate with 1.2 g kg⁻¹. According to the recommendations of Silveira *et al.* (2000) all periods were below adequate.

Even applying 280 kg ha⁻¹ of soluble P₂O₅ close to the seedling over a year, this practice was not sufficient to change leaf concentrations, with the first 24 months being the period of lower concentration, increasing concentrations over time, behavior that was also found by Rocha *et al.* (2016) for *Eucalyptus grandis*. This condition was not expected given the low availability of this nutrient, adding the characteristics of the Oxisols used, with high acidity and high degree of weathering (TABLE 1) the responses should be high. Thus the critical ranges established in the literature were not sufficient to determine the critical leaf level for the conditions of the studied sites.

Although no changes in leaf concentrations were observed, the application of soluble phosphate enabled visible dendrometric growth in the field in the early period, reinforcing the function of early growth accelerator in *Eucalyptus dunnii* (DIAS *et al.*, 2017). However, as time went on, the treatments became evenly matched, indicating that the trees began to absorb more P from soil. The possibility is that the P came from the reactive natural phosphate applied during subsoiling. Since, it is well known this P source has low release capacity.

Potassium - At Jaguariaíva site at 6 months all treatments of experiment K maintained values between 9 and 10 g kg⁻¹, considered adequate for Silveira *et al.* (2000) and Gonçalves *et al.* (2011). After this period only the fertilized treatments with 280 kg ha⁻¹ had values considered adequate for Gonçalves (1995). In Ventania, the mean K concentrations at 24 and 36 months were adequate according to values presented by Gonçalves (1995). Even in the Ventania site with contents higher than Jaguariaíva, these were not enough to reach the adequate level for Silveira *et al.* (2000). This critical range proposed by Silveira *et al.* (2000) is probably elevated for local conditions, since other works with leaf contents between 5.00 and 8.50 g kg⁻¹ had good growth for *Eucalyptus urograndis* (JESUS *et al.*, 2012; SILVA *et al.*, 2013).

Critical K levels for some *Eucalyptus* species planted in Brazil should be further studied and need to be defined by species, clone and hybrid. Perhaps the concentrations indicated in the publications are high, or are for tropical climatic conditions or even with water restrictions (BIAGIOTTI *et al.*, 2017).

The K deficiency leaf symptom was observed from the sixth month in the absence of the element in Jaguariaíva, presenting concentration 3.1; 4.7 and 4.0 g kg⁻¹ at 12, 24 and 36 months, respectively. Thus, values close to 3 to 4 g kg⁻¹ represent lower limit for the occurrence of deficiency. Still, the only visual analysis of K symptom may not be the most appropriate, since it manifests itself in very low concentrations, and the detrimental effect on growth is slower than the onset of the symptom, since only from the 24th month onwards decrease in growth (BASSACO *et al.*, 2018).

Increasing K requirement as reforestation age has been indicated by several authors (BATTIE-LACLAU *et al.*, 2014), and part of this required K may come from internal retranslocation at a rate of 40 to 75%. (LACLAU *et al.*, 2010). However, when the soil and plant are at low levels of K, it was unable to maintain concentrations equal to the highest doses. This explains the onset of response to K application after 24 months. In addition, with this high internal retranslocation there is a slight decrease of K in the leaves, consistent with the results found by Rocha *et al.* (2016). Therefore, it is important to define deficiency levels under local conditions, because even under deficiency the plant took a long time to reduce growth, and even with below adequate levels according to the literature eucalyptus in both places had excellent growth (65 m³ ha⁻¹ year⁻¹).

Temporal variation of macros, micronutrients and aluminum in leaf tissue over time

Calcium contents at both sites at 12, 24 and 36 months are within the appropriate level for Gonçalves (1995), while for Silveira *et al.* (2000) at 12 and 24 are adequate and the reduction in concentration at 36 months leaves were considered deficient. Decreased leaf contents were expected, as most absorbed Ca is not retranslocation to the leaves, but required for tree trunk and bark formation (LACLAU *et al.*, 2000; HAWKESFORD *et al.*, 2012).

Magnesium was at an adequate level for Silveira *et al.* (2000) and Gonçalves (1995) in all periods and sites evaluated. The importance of liming in the supply of Ca and Mg in deficient and acidic soils is highlighted (Rodrigues *et al.*, 2016), seeing that the levels of the soil were very low. We can observe that the leaf contents increased with age and that this is one of the organs that accumulates more of this nutrient, an expected situation due to its enzymatic and structural functions in chlorophyll (LACLAU *et al.*, 2000; HAWKESFORD *et al.*, 2012).

Regarding the concentrations of the main micronutrients and other evaluated elements, it was observed in the literature that most of the sequence found is Mn > Fe > B > Zn > Cu (SILVEIRA *et al.*, 2000; ALBAUGH *et al.*, 2015). While in the present work the sequence found was Mn > B > Fe > Zn > Cu, a sequence that probably changes due to boric acid fertilization. High B values suggest that it may decrease the dose used in fertilization or even split, thus avoiding such high levels. However, at 24 months the sequence changes to Mn > Fe > B ≥ Zn > Cu, keeping the literature standard.

The separation of micronutrients and other elements based on concentration greater than 100; between 10 and 5; 5 and 1; and less than 1 mg kg⁻¹, varied according to plant age and forest site. The Mn was always above 100 mg kg⁻¹ at the different ages evaluated, values also observed by Jesus *et al.* (2012) for *Eucalyptus* aged 2.5 years and Albaugh *et al.* (2015) on trees between 0 and 3 years. For Fe and B, the concentrations ranged from 100 mg kg⁻¹ or higher, being influenced by the plant age, the older, the lower the observed concentration. This indicates a probable dilution effect due to the maximum increase in leaf area up to the 36th month (MELO *et al.*, 2016). The opposite effect occurred with Zn and Cu which over time increased their concentrations and reached the classes of 10 to 50 mg kg⁻¹ and 1 to 10 mg kg⁻¹, respectively.

Regarding the Cu content in the leaves, in all experiments a level considered low, less than 3.5 mg kg⁻¹ was observed. The same behavior was observed for Zn, but closer to the 7 mg kg⁻¹ deficiency limit. This condition is expected in extremely weathered soils developed under sandstone rocks. Although the observed growth is high and has no apparent symptoms of Zn and Cu deficiency, it is recommended, based on the data, their basic fertilization. Iron was within the considered range of 63 to 200 mg kg⁻¹ (SILVEIRA *et al.*, 2000; GONÇALVES, 1995).

The B values at the age of one year were 4 times above the range of 30 to 50 mg kg⁻¹, proposed by Silveira *et al.* (2000) indicating a rapid availability of B in a short period and decrease over time, indicating the need to reduce the applied dose and installment, although no symptoms of toxicity were observed. The low residual effect due to the high B leaching capacity in the soil may be related to the rapid decrease of B content after application.

The Mn concentration obtained in the first evaluation was above the considered adequate limit of 840 mg kg⁻¹ by Gonçalves (1995) and Silveira, but within the value established by Dell *et al.* (2002), which goes up to 2,200 mg kg⁻¹. The values can be considered normal as the plants did not show apparent symptoms of Mn toxicity and the growth was adequate (BASSACO *et al.*, 2018). Reinforcing the indication of normality, Harguindeguy *et al.* (2018) found values between 300 and 5,083 mg kg⁻¹ in *Eucalyptus* seedling leaves developed under controlled oxygenation condition and Mn availability, without being able to associate this variation with growth of the two studied clones.

The low soil pH and the decomposition of pine litter can explain this high concentration of Mn (VIERA; SCHUMACHER, 2010). These factors increase the availability of Mn in the soil, as well as the solubilization effect caused by organic acids of organic matter, evidenced by the higher Mn concentration in the Ventania site. As for Mn, Al presented high concentration and can be associated with high acidity and abundance of available form, m % > 80 %, reinforcing the overview of high resistance of eucalyptus to soil acidity.

Potassium deficiency test

We observed in the potassium deficiency test that the results of leaf analysis were not consistent to indicate K deficiency, because even with severe signs of the symptom the leaf contents were statistically similar to leaves without symptoms, with only 5% variation between them. This information reinforces that leaf K content depending on the site may not be sufficient to diagnose nutrient deficiency (SILVA *et al.*, 2013; ALBAUGH *et al.*, 2015; BATTIE-LACLAU *et al.*, 2014).

Another behavior observed in potassium deficient plants was the reduction of leaf life, shorter leaf area and symptoms in leaves proximal to the trunk, corroborating with Laclau *et al.* (2009).

CONCLUSION

- N and P applied nutrients had no effect on leaf concentrations over 36 months. The application of K changed leaf concentrations at 24 and 36 months, but at 12 months even with leaf symptoms, there was no change in concentration
- Time is an important factor in determining the concentration of the elements and is therefore crucial for determining age and establishing critical ranges of the elements.
- Critical ranges for N, P, and K were not sufficient to determine the critical level of these elements, and were often overestimated values for the study condition.
- Finally, we can observe that *E. urograndis* is highly resistant to Al and Mn; even at high leaf concentrations, they obtained good growth.

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