

Article

Chemical and Kraft Pulping Properties of Young Eucalypt Trees Affected by Physiological Disorders

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Abstract: This study evaluated how *Eucalyptus* physiological disorder (EPD) affects wood quality and pulping performance. Although research advances have been made in forest management and tree improvement programs for eucalypt plantations, some areas of Brazil are still subject to abiotic stress, mainly due to atypical climatic patterns. Tree growth is affected by abiotic stress, and this can change the wood properties, which influence the pulping process. The *Eucalyptus* trees used in this study were three-and-a-half-year-old hybrid clones. In order to evaluate the impact of physiological growth disorder on the wood, trees were selected with higher and lower levels of symptoms caused by EPD. First, the density, chemical composition, and variables of the pulping process of each of these woods were compared. The higher levels EPD symptoms resulted in poorer wood quality for pulping. To reduce the negative impact of the pulping process, reference woodchip samples from the industrial process were mixed with these wood samples and evaluated again. The results show that EPD negatively affected the wood quality required for pulp production when trees from higher stress conditions formed wood with more extractives (60% greater) and 9% more of lignin content. Thus, the amount of reagent used was increased and the pulp yield decreased. One solution to minimize the problem is to combine the woodchips from higher EPD trees and stands (20%) with non-EPD-affected chips (80%). Thus, it appears that affected wood requires special management attention in the context of pulp production.

Keywords: tree plantations; abiotic stress; growth disorders; wood quality



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1. Introduction

In Brazil, the forest plantations of the *Eucalyptus* genus play an essential role in supplying raw material to different industrial uses, for example, pulp and paper, energy, and panel and sawn wood. According to the most recent 2021 Annual Report of the Brazilian Tree Industry (IBA) [1], approximately 78% of the 9.5 million hectares of planted forests in Brazil are occupied by eucalypt trees, representing the most important fiber source in the national pulp industry. This remarkable performance of the *Eucalyptus* genus in Brazil is explained by many studies dedicated to tree breeding and forest management in the past 70 years [2].

Despite all of the advances in tree breeding and forest management focusing on faster growth, eucalypt trees must deal with complex growth-related interactions. In general, abiotic stresses are external environmental factors (e.g., heat, drought, high salinity, and cold) that are detrimental to the establishment and/or growth. These often manifest as growth reductions, changes in the chemical composition (e.g., lignin and extractives), and fewer natural defenses [3–5]. As this condition firstly affects the growth of the trees, it also

highlights a trade-off of concern between stand productivity and wood quality. Abiotic stresses have been affecting some *Eucalyptus* plantations in Brazil for the past two decades. This adverse condition is called *Eucalyptus* physiological disorder (EPD). In 2008, forest technicians from Suzano S.A. (formerly Fibria Celulose S.A.) identified, for the first time, symptoms and signs of this new disorder in regions located at the north of Espírito Santo State and the extreme south of Bahia State, Brazil. EPD is a disease of abiotic etiology, which causes the loss of apical dominance, reduced growth, and deformation in the form of minicankers on the stems and the apical meristem. EPD is also characterized by the occurrence of the death of the leaf fall, crown forking, increased lateral sprout, and cracks and exudates present in the bark. Studies carried out in recent years (unpublished data) have shown that the disease is caused by the low adaptability of certain *Eucalyptus* clones to particular climate conditions.

Recently, researchers have focused on understanding the plant responses to climate change, the adaptability of eucalypt clones, as well as wood productivity [4,6–9]. Considering that the cause of this disorder appears to be directly related to the changes in environmental condition profiles, the effects of different environments on eucalypt wood formation and properties have been studied [10–14]. However, only a few studies have been related to growth disorders and their effects on the wood quality required for pulping production [15–18].

To predict the pulping performance of wood clones, properties such as the specific gravity and chemical composition must be assessed [19,20]. They are considered essential since they directly relate to the processing efficiency, chemical reagent consumption, and pulping yield. Câmara et al. [17,18] investigated the influence of physiological disorders on eucalypt pulp production in trees of two ages (three and seven years old). Overall, the results indicated the disorders affected the chemical composition and pulping variables but did not affect the specific gravity (density). The opposite result was reported by Jardim et al. [16] studying hybrid clones of *Eucalyptus grandis* × *Eucalyptus urophylla* of eight years old. The authors recorded a reduction of 6.7% in the density of the clones sensitive to EPD.

Although there is widespread use of *Eucalyptus* genus in forest plantations in Brazil, information about the effects of abiotic stress, herein EPD, on wood properties for pulping production is incipient. Therefore, understanding the wood quality of young eucalypts grown under EPD conditions will provide valuable and necessary information in regard to the subsequent pulping process of the wood as well as a response to the forest breeding program to assist with the selection of the clones. In this context, the objectives of this study were to understand how EPD affects the physical, chemical, and variables of the pulping properties of a young clone of *Eucalyptus grandis* × *Eucalyptus urophylla* and to determine an appropriate woodchip mix ratio in order to minimize the negative impact of EPD wood on the pulping process.

2. Materials and Methods

To perform this investigation, two nearby *Eucalyptus grandis* × *Eucalyptus urophylla* plantation stands of three and a half years old were selected. These eucalypt plantations were established in Teixeira de Freitas, south of Bahia State, Brazil. The site studied is located at the coordinates of 39°54′01″ south and 17°29′17″ west, with a mean altitude of 120 m, mean annual precipitation of 1112.8 mm, mean temperature of 23 °C, and relative air humidity of 79%.

These two stands were considered representative for the research herein, as they exhibited trees with a visual contrast of abiotic stress (EPD) affecting them. They were colloquially named lower stress (Figure 1A) and higher stress (Figure 1B).



Figure 1. Visual effects of *Eucalyptus grandis* × *Eucalyptus urophylla* clone plantations under lower EPD symptoms (A) and higher EPD symptoms (B); field sampling (C–F).

To perform this study, nine trees from each level of EPD symptoms were selected and harvested (Figure 1C–F). The trees from the lower level of symptoms showed a mean commercial height of 16.8 m and a diameter breast height (DBH) of 14.5 cm. The trees from the higher level of stress conditions showed a mean commercial height of 11.2 m and a DBH of 11.9 cm. Each trunk of each eucalypt tree was transformed into 1-m-long bolts from base to top, transferred to the laboratory yard, and, one week later, chipped with a woodchipper).

In laboratory conditions, the woodchips were classified, dried, and stored in labeled plastic bags for subsequent analyses. The specific gravity (density), one of the best wood quality parameters for pulp production, was determined using the immersion method described in NBR 11941 [21]. For chemical analysis, the dried woodchips were ground in a Wiley mill, and the dust produced was classified with 40 to 60 mesh sieves. The chemical composition of the woodchips included ethanol–toluene extractives, acetone extractives, lignin, and pentosans (see Table 1, with laboratory procedures).

Table 1. Standards used for chemically analyzing the wood of *Eucalyptus grandis* × *Eucalyptus urophylla*.

Analysis	Procedure
Ethanol/toluene extractives	TAPPI T204 cm 97 (solvent extractives from the wood pulp) [22]
Acetone extractives	TAPPI T280 pm 99 (acetone extractives of wood and pulp) [23]
Lignin	TAPPI T222 cm 06 (acid-insoluble lignin in the wood and pulp) [24]
Pentosans	TAPPI T223 cm 10 (pentosans in the wood and pulp) [25]

Assuming the hypothesis that higher levels of EPD symptoms negatively impact pulping, four more treatments were designed (see Table 2). Treatments 1 and 2 represent the trees under lower and higher EPD levels of symptoms (contrasting). Treatment 3 refers to woodchip mixes used at the company laboratory, which simulated the wood composition used in the industrial process. In addition, to understand the impacts of EPD on pulping, Treatments 4 and 5 were created. Both treatments were supplemented by Treatment 1, and the cooking variables (alkali charge (%), temperature (°C), residual alkali (g/L), and screened yield (%)) were determined.

Table 2. Composition of the treatments—wood samples from different levels of stress and mixture used in pulping process.

Treatments	Description
1	Wood under lower stress
2	Wood under higher stress
3	Wood mix (composition of different woods)
4	80% of the treatment 3 + 20% of the treatment 2
5	20% of the treatment 3 + 80% of the treatment 2

The cooking was carried out in an M&K 10-L batch laboratory digester system. It has a capacity of 1200 g of dry wood chips per batch, and we have a standard of using 800 g of dry eucalypt chips per batch. A liquor-to-wood ratio of 4:1 was used for all batch cooks. To determine the cooking variable values, the active alkali charge on the pulping liquors was varied according to the necessity for generating a standard Kappa number. Thus, the alkali charge was adjusted to produce a kraft pulp kappa number of 18 ± 1 . The residual alkali was fixed between 1 and 5 g/L, the liquor sulfidity was 35 ± 1 , and the overall cooking time was 290 min.

After delignification, the kraft pulp was screened and the waste was separated, dried, and weighed. To quantify the waste, the percentage of dry weight for the waste and woodchips that supplied the waste was calculated. To determine the pulp screened yield (SY), the difference between the raw waste and waste content was calculated.

The experimental design used in this investigation was completely randomized. The data were analyzed for normality using the Shapiro–Wilk test, and analysis of variance (*F*-test) and Tukey’s test at a 5% significance level were used to evaluate and compare the mean parameters considered in this study.

3. Results and Discussion

In this research, the physical–chemical wood properties (Table 3) and the cooking variables (Table 4) were determined to understand the real effect of EPD on eucalypt trees that grew in these conditions. Additionally provided is an estimate of the maximum percent of stressed wood that can be added to industrial processing (Table 5). For that, nine trees at each level of EPD symptoms were collected, sampled, and analyzed.

Table 3. Mean values of the woodchip specific gravity, chemistry, and pulping of *Eucalyptus grandis* × *Eucalyptus urophylla* trees under two growth conditions.

Symptom Level	SG	EXT (%)	EXA (%)	LIG (%)	PEN (%)
Low	0.488	1.1 *	1.0 *	28.26 *	15.61
High	0.491	1.8	2.0	30.82	16.29

SG: Woodchip specific gravity; EXT: Ethanol/toluene extractives; EXA: Acetone extractives; LIG: Total lignin; PEN: Pentosans. * Significant at 5% using the *F*-test.

Table 4. Mean values of the wood cooking variables of *Eucalyptus grandis* × *Eucalyptus urophylla* trees under two EPD levels.

Symptoms Level	AC (%)	T (°C)	RA (g/L) *	SY (%)
Low	16.0	147	1.73 *	55.8 *
High	18.0	148	3.02	51.5

AC: Alkali charge (%); T: Temperature (°C); RA: Residual alkali (g/L); SY: Screened yield (%). * Significant at 5% using the *F*-test.

Table 5. Mean values of the wood pulping variables of *Eucalyptus grandis* × *Eucalyptus urophylla* under higher stress and a cooking mix.

	Treatments	AC (%)	T (°C)	RA (g/L)	SY (%)
3	Wood mix	15.5 c	147	1.2 b	54.7 a
4	80% of the wood mix +	16.5 b	146	1.4 b	54.3 a
	20% of the wood with higher EPD symptoms				
5	20% of the wood mix +	18.5 a	148	4.2 a	52.1 b
	80% of the wood with higher EPD symptoms				

AC: Alkali charge (%); T: Temperature (°C); RA: Residual alkali; SY: Screened yield (%). Mean values followed by the same letter are not significantly different (Tukey test, $p > 0.05$).

The woodchip density was not significantly different between these two conditions, as shown in Table 3, using the *F*-test (ANOVA) at the 5% significance level (see Table 3). Thus, EPD did not affect the specific gravity of the eucalypt trees studied herein. The same result was found by Câmara et al. [17,18] investigating the influence of physiological disorders caused by abiotic stress in *Eucalyptus grandis* × *Eucalyptus urophylla* at two ages (three and seven years old). In contrast, a previous study reported on the influence of EPD on the specific gravity of eight-year-old *Eucalyptus grandis* × *Eucalyptus urophylla* clones [14]. There, the authors found a reduction of 6.7% for the clones sensitive to EPD and 4.8% for the clones tolerant to EPD.

The mean values of the ethanol/toluene (%) and acetone (%) extractive contents were significantly different using the *F*-test (ANOVA) at a 5% significance level. The mean values for hemicelluloses represented herein by pentosans were not significantly different using the *F*-test (ANOVA) at a 5% significance level.

The EXT (%) content increased by more than 60% for wood with higher symptoms of EPD. A significant increase was also observed for the EXA (%) content, which was twice the level as that in the wood with lower symptoms level of EPD, that is, it was increased by 100%. The same tendency was reported in the literature [16]. However, Câmara et al. [17,18] found the opposite behavior for these compound contents. In general, secondary metabolites are influenced by environmental changes, site, provenance, and the like, and they are essential for plant defense and survival [26,27]. This alteration of wood chemical compounds seems to be an important defense response of the plant against insects and diseases [27].

According to Berini et al. [28], the secondary metabolism of woody plants is influenced by combinations of varying abiotic factors, that is, this compound formation occurs via a range of complex and highly variable responses. The *Eucalyptus* genus has the capacity and ability to produce specialized metabolites (e.g., terpenes, formylated phloroglucinol compounds, phenolics, and cyanogenic glucosides) to play this seesaw, moderating the interaction between environmental changes and the plasticity to adapt [29,30].

Following the same tendency of extractives, the lignin content values were affected by the EPD symptoms and they increased by over 9.0% in the wood with higher EPD symptoms in the study herein. Increased lignin contents of hybrid *Eucalyptus grandis* × *E. urophylla* trees under abiotic disorder stress has been reported in the literature [16,18]. The authors found increases of 3.2%, 4.5%, and 5.0%. However, Câmara et al. investigated the same hybrid four years older than in the current study and found no significant influence on the total lignin content. The wood formation is considered highly plastic and involves the dynamic integration of environmental signals, consequently affecting the lignin deposition and content [31,32]. The reason for this is that lignin biosynthesis plays a crucial role in cell formation and this chemical compound is affected by abiotic stress [3,27].

It is important to note that the extractives and lignin contents found in this study are considered high because the wood samples included trees with the same genetic material, planted in the same surrounding site, and at the same age. However, it is also important to consider that the wood in this study was just three and a half years old, and thus, very young raw material.

To determine the cooking variables, the cooking standard for the Research Center of the Suzano Company was set at an 18 ± 1 kappa number and a residual alkali between 1 and 5 g/L. The mean values for the pulping variables of the eucalypt wood under two symptom levels of EPD are shown in Table 4.

This increased alkali charge meant that the delignification of the wood with higher EPD levels produced a lower screened yield (drop of 4%) and, consequently, produced higher apparent specific consumption in the industry. The wood from higher EPD trees consumed more reagents and needed a superior temperature compared to the wood grown with lower EPD symptoms. In cases where the specific gravity and pentosans did not appear to influence the cooking behavior, the higher levels of extractives and lignin contents of EPD wood became influential. These results are considered problematic because they could cause gaps in the supply of wood in the company's pulping process. The results of higher values of extractives and lignin contents produced by the EPD indicate the need for harsher pulping conditions to reach a pre-established kappa number [16]. The pulp yield is considered one of the primary parameters in evaluating wood quality because it is directly related to the anatomical and chemical structures of the wood, alkali charge demand, digester production, and solids load for the recovery boiler [33].

To decrease the negative effect on the pulping process caused by woodchips prevent from trees with higher EPD symptoms, a composition of different woodchip specimens was prepared to create new treatments and to verify the cooking variables. Therefore, the woodchip sample composition ("mix") that was representative of the factory supply was used and supplemented with woodchips from the wood with higher EPD symptoms. Ratios of 80% and 20% were used. The values generated for the cooking processes are shown in Table 5.

According to the results, the wood mix displayed excellent performance in the cooking process because it had the highest screened yield value and likely the lowest apparent specific consumption. However, when this wood mix was supplemented with 80% of the material with higher EPD symptoms, the reagent consumption increased from 15.5% to 18.5%. This treatment produced one of the worst results for the cooking processes (see Table 5), with a 52.1% screened yield. When the dose of the woody material with higher EPD symptoms decreased to 20%, the alkali consumption also decreased, and the cooking process had a 54.3% screened yield. Therefore, a maximum dose of not more than 20% of the woody material with higher EPD symptoms is recommended for use in the manufacturing process. Based on the authors' knowledge, this work represents the first attempt of its kind to assess the wood properties in the contrasting levels of EPD symptoms and apply it to simulate the cooking behavior in a real situation of eucalypt wood supply in the pulp industry.

4. Conclusions

The density (SG), chemical, and cooking variables of young clonal *Eucalyptus grandis* × *E. urophylla* trees were evaluated at two contrasting symptoms levels of Eucalyptus Physiological Disorder (EPD), and it was found that the EPD symptoms did not influence specific gravity and pentosans, but negatively affected extractives and lignin contents.

The results of the study show that EPD needs particular attention, especially in regard to the provision of wood with the quality required by the pulp mill. The highlight of the study is the impact of stress on the wood regardless of the fact that the trees were of the same clone, the same age, and planted with the same management, but planted in adjacent stands.

In such instances, it is advised that a dosage of less than 20% of the woody material with greater EPD symptoms is utilized in the pulping process. As a strategy for a forest improvement program, it is important that the genotypes used are less sensitive to environmental variations. In addition, understanding how the *Eucalyptus* trees respond to physiological disorders in wood formation could also be a very important tool to use in transgenic strategies to create EPD-tolerant clones.

Further studies should evaluate the ratio between syringyl and guaiacyl to understand the relationship between physiological disorder levels and lignin quality.

Author Contributions: B.P.R. conducted the field experimental investigations, performed the lab experiments, collected and analyzed the data, curated the data, and prepared the manuscript; B.J.D. and R.G.M. gathered the resources, designed the study, supervised the experiments at all stages, and provided critical feedback, and reviewed and edited the manuscript; J.T.d.S.O. supervised the study, gathered the resources, and reviewed and edited the manuscript; G.B.V. reviewed and edited the manuscript. All authors have read and agreed to the published version of the manuscript.

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