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# PHYSICAL AND CHEMICAL TRAITS IN FALSE HEARTWOOD FOR TWO *POPULUS* HYBRIDS

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## ABSTRACT

A comparative analysis was performed to determine the density, moisture content, shrinkage, pH, and concentration of potassium and calcium in samples of false heartwood and sapwood from two commercial poplar hybrids. The results showed that the calcium concentration is on average three times higher in false heartwood than in sapwood. In turn, the potassium concentration is on average five times higher in false heartwood than in sapwood. The pH increased from slightly acidic in sapwood to slightly basic in false heartwood. The moisture content is markedly higher in false heartwood than in sapwood. However, density and shrinkage do not differ between the two types of wood. A chemometric model analysis was used to differentiate between the two types of wood by using near infrared reflectance spectroscopy (NIRS) with multivariate analysis. The NIRS-implemented method demonstrated efficiency to discriminate between both types of wood.

KEYWORDS: False heartwood, poplar hybrids, chemical traits, calcium, infrared spectrometry.

## INTRODUCTION

The presence of false heartwood in poplar wood is a significant problem that reduces its industrial use. False heartwood appears at the center of the poplar stems and has different organoleptic properties than the rest: it is slightly darker in fresh wood, a relatively intense smell (rancid) and notably higher moisture content than the area around it (Wallin 1954, Nakada 2014). The formation of false heartwood is attributed to the presence of anaerobic bacteria made up of *Erwinia* sp, *Methano bacterium* sp or *Clostridium* sp., which primarily attack standing trees (Schink et al. 1981, Sakamoto and Kato 2002). For its industrial use, the wood with false

heartwood dries at a much slower rate that regular wood (De Boever et al. 2011). Because of this, when wood with false heartwood is mixed with regular wood in the drying process, it does not attain an equilibrium moisture content in the planned time. Consequently, the poplar processing industry disqualifies the log area with false heartwood, which limits its use and affects the profitability of wood production (Alkan et al. 2007). Ward and Pong (1980) provide a general description of false heartwood for different forestry genera, mainly: *Populus, Platanus, Salix, Abies* and *Tsuga*. According to the described evidence it seems that certain bacteria are part of a normal microflora in the wood but are inactive until a certain change occurs and the wood starts to form a substrate favorable for their development, where they subsist on tree sap (Nakada 2014, Ward and Pong 1980). (Johansson and Hjelm 2013) Studied the internal distribution and shape of false heartwood inside the *Populus* stem and calculated predictive equations on the diameter and volume of false heartwood in terms of DBH. The researchers found that the false heartwood has a conical form to inside the stem, representing a proportion of the volume depending on average tree height; up to 13% in specimens with a DBH of 150 mm and up to 22% in specimens with a DBH of 500 mm.

The bacteria responsible for false heartwood are anaerobic, which explains their presence in the innermost part of the stem. This study speculates that the bacterial complex degrades the pectin in the primary cell wall and middle lamella, resulting in an increase in pressure of certain gases in the trunk, such as carbon dioxide, hydrogen, methane, nitrogen and hydrogen sulfide (Ward and Pong 1980, Jeremic et al. 2004). The primary physical and mechanical properties would not be altered in false heartwood, except permeability and pH. Regular wood has a slightly acidic pH, however false heartwood has a slightly basic pH (Poblete et al. 1991, Walter 1993). The bacteria reduce the permeability of the wood by blocking the lumens, primarily due to the presence of polysaccharides (Ward and Pong 1980, Walter 1993, Xu et al. 2001). Regarding chemical differences, through the use of X-ray fluorescence, (Smith et al. 2014) recently detected a marked reduction in the potassium concentration in healthy sapwood (active sapwood) from *Salix alba* over the more central area (inactive sapwood), which presents an incipient pathogenic attack. However, further towards the center of the stem, this trend reverses, drastically increasing the concentration in the area closest to the pith (central decay). This shows that a detailed analysis of certain ions, at a level of growth rings, could potentially provide a chemical differentiation for pathogenic, physiological and anatomical effects on trees.

When wood is chemically transformed, whether into cellulose pulp or biomass, false heartwood is not a significant problem. On the other hand, when wood is used in solid form, false heartwood is a problem, as it reduces the degree of usability (De Boever et al. 2011, Smith et al. 2014). This highlights the importance of identifying, on the one hand, the magnitude of false heartwood based on the soil and climatic traits in a given site, and on the other, clonal dependencies and interrelationships between these factors and the quality of the final products (Johansson and Hjelm 2013). For these purposes it would be useful to have non-destructive means of predicting the magnitude of false heartwood in stands through quick and easily implementable analyses. This could involve the analysis of certain traits of false heartwood that are easy to measure, without recurring to tree felling or waiting until harvest (Tsuchikawa et al. 2003).

The purpose of this study is to document the differences between sapwood and false heartwood in some of their physical (moisture content, density and shrinkage) and chemical (extractives, acidity, ashes, and concentration of potassium and calcium ions) traits. In conjunction with this, the study uses chemometrics to differentiate between each type of wood via diffuse infrared reflectance spectrometry in conjunction with multivariate statistical analysis. This latter method establishes a quick and reliable mechanism for determining the presence of false heartwood in processed wood from *Populus* hybrids.

## MATERIAL AND METHODS

### Sample preparation for measuring physical properties

A total of 6 wood discs from 6 different trees of *Populus deltoides* × *Populus nigra* 'NNDV' and 'Luisa Avanzo' were collected from plantations located in the district of Retiro, Maule Region, Chile. Both stands with a planting density of 278 trees $ha^{-1}$ were 14 years old at the time of their study and had identical forest management. A disc of 20 cm thick was obtained from first log of 70 cm at the base of each tree. For moisture content, density and shrinkage, we studied the 3 specimens of each hybrid separately to estimate statistical differences between hybrids. Four samples of 2.5 × 2.5 × 10 cm were taken in a radial, tangential and longitudinal direction, respectively, and moisture content (NCh 176/1 Of. 1984), basic density (NCh 176/2 Of. 1986), and shrinkage coefficients (NCh 176/3 Of. 1984) were determined for each. For determining pH, total extractives and extractives soluble in sodium hydroxide, the study analyzed two mixed samples from the three specimens from each hybrid. Samples were pulverized in a Retsch mill, model SM 100, sieved in a 40 mesh screen and oven dried at 103°C. These samples determined extractives and solubles in a 1% NaOH solution separately for each specimen. Extraction of extractives began in an ethanol/cyclohexane solution, as opposed to ethanol/benzene, in a 1:2 ratio for 6 hours (ASTM D1107-84), followed by pure ethanol for 4 hours (Browning 1967) and finally hot water for 2 hours (ASTM D1110-84). The extractives ratio was calculated based on the initial anhydrous mass. Solubles in 1% NaOH were obtained by keeping the solution at 97 – 100 °C for one hour and weighing the insoluble residue (Browning 1967).

### NIR spectroscopy and data analyses

Shavings from sapwood and false heartwood areas were milled in the Retsch mill, model SM 100, into sawdust. The study reviewed the 3 specimens from each hybrid separately. Sawdust was oven dried at 103°C until reaching constant mass. The diffuse infrared reflectance spectrum was recorded with Unity equipment, model 2200, which covered a spectral range from 1100 to 2200 nm with a spatial resolution of 1 nm. The scan time was 150 seconds, in which time the device internally averaged 120 spectral measurements. This was followed by a Principal Components Analysis (PCA) with a Calstar 2.00 program (Sensologic - Germany) to distinguish between false heartwood and sapwood. The study determined the spectral ranges that were most relevant for this discrimination, using the loading matrix from the PCA analysis.

### Chemical analyses

The study used atomic absorption (AA) spectrophotometry to determine the potassium and calcium content. Chemical treatment involved weighing 1.0 g of sample sieved in a porcelain crucible and calcined slowly up to 500°C over 6 hours. The crucibles were left to cool to room temperature, at which point, 2 mL of bidistilled water and 5 mL of 5 molar nitric acid were added under a hood. The solutions were heated on a heating plate and constantly stirred until almost dry. The solutions were then filtered through systems with 0.45 μm porosity. The filtrate had a final volumen of 50 mL with bidistilled water (Walinga et al. 1995, Karla 1998). The study made AA determinations with a flame technique (air/acetylene), using a Unicam brand spectrophotometer, model 969. The instrument was calibrated with standard solutions from Fisher Scientific International, Inc. The wavelenth was 766.5 nm for potassium and 422.7 nm for calcium. A single sample was determined per tree.

## RESULTS AND DISCUSSION

Tab. 1 presents the comparative results for false heartwood and sapwood in both hybrids. Total extractives are similar, around 2%, in both sapwood and false heartwood in both hybrids. Extractives soluble in sodium hydroxide were also similar, around 20% in sapwood and false heartwood for both hybrids. An increased solubility in sodium hydroxide is associated with a higher ratio of molecules with lower molecular mass, which is related to a greater susceptibility to wood decay fungi, or rather a fungal deterioration in the cell wall (Yilgor et al. 2013). In fact, for *P. balsamífera* and *P. tremuloidesa* solubility in sodium hydroxide of around 10% had previously been determined for sapwood and slightly higher, around 14% for false heartwood (Micko 1987). In our case, no increased solubility in sodium hydroxide is detected in false heartwood and therefore this does not lead to fungal deterioration. The slightly acidic pH in sapwood, which is considered normal for wood, becomes neutral to slightly basic in false heartwood, which is indirect evidence of bacterial colonization (Walter 1993). In both hybrids, the moisture content in fresh wood is significantly higher in false heartwood than in sapwood (p-value< 0.01). The 'Luisa Avanzo' hybrid presented a basic density of 369 $kg \cdot m^{-3}$., significantly higher than the 'NNDV' hybrid, with 345$kg \cdot m^{-3}$. Both hybrids showed significant differences in basic density between sapwood and false heartwood. False heartwood shows a lower density than sapwood. This decrease can be explained by the formation of juvenile wood. On the other hand, the study found no significant differences in shrinkage between false heartwood and sapwood for both hybrids. Shrinkages were measured at equilibrium moisture content (EMC) of 10.8 ± 0.2% after the specimens had passed through a reconditioning process that lasted for 2 hours at 100°C and 100% relative humidity. In this process, the study saw the cell wall collapse in two false heartwood specimens. In one of these, the volumetric collapse reached 11.5% ('NNDV' specimen) and in the other the volumetric collapse was 5.0% ('Luisa Avanzo' specimen).

*Tab. 1: Average values of physical and chemical traits for false heartwood and sapwood.*

| | Number of samples | False heartwood 'Luisa Avanzo' | Sapwood 'Luisa Avanzo' | False heartwood 'NNDV' | Sapwood 'NNDV' |
|---|---|---|---|---|---|
| Total extractives (%) | 2 | 1.6 (0.1) | 1.9 (0.1) | 2.3 (0.1) | 1.7 (0.1) |
| NaOH soluble (%) | 2 | 19.6 (0.6) | 20.5 (0.2) | 22.5 (0.1) | 19.6 (0.3) |
| pH | 2 | 7.83 (0.04) | 5.83 (0.05) | 8.35 (0.39) | 5.72 (0.05) |
| Oven dry basis moisture content (%) | 4 | 181 (29) | 100 (2) | 170 (48) | 75 (5) |
| Basic density ($kg \cdot m^{-3}$) | 4 | 352 (9) | 386 (11) | 335 (5) | 355 (12) |
| Tangential shrinkage at 10% (%) | 4 | 6.0 (1.6) | 5.4 (0.2) | 4.9 (0.3) | 5.1 (0.3) |
| Radial shrinkage at 10% (%) | 4 | 2.3 (0.5) | 2.3 (0.3) | 2.2 (0.4) | 2.0 (0.7) |

Note: the values in parentheses refer to standard deviation.

Based on Fig. 1, the ratio of total ashes increases 3.5 times, from an average value of 0.45% in sapwood to an average value of 1.58% in false heartwood. These values coincide with the values determined by (Micko 1987), ranging from 0.6% to 0.9% for sapwood and 1.2% to 1.9% for false heartwood, in two different *Populus* species. The ratio of ashes in false heartwood is higher in the 'NNDV' hybrid than the 'Luisa Avanzo' hybrid (p-value = 0.031).

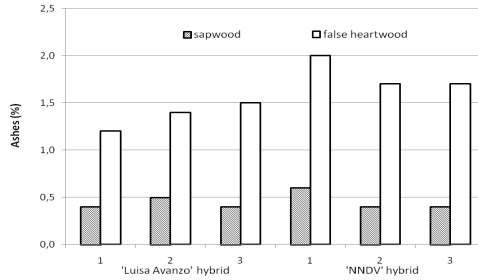


*Fig. 1: Result of ashes for false heartwood and sapwood in two Populus hybrids.*

In terms of the chemical composition, the potassium concentration (Fig. 2), increases by a factor of 5, from an average of 605 $mg \cdot kg^{-1}$ in sapwood to 3186 $mg \cdot kg^{-1}$ in false heartwood. These results coincide with those determined by (Fukazawa et al. 1985), who studied *Populus* maximowiczii specimens and measured average potassium concentrations of 300 $mg \cdot kg^{-1}$ in sapwood and 1350 $mg \cdot kg^{-1}$ in false heartwood. However, these are lower than those found by (Janin and Clément 1972), who studied *Populus* trichocarpa 'Fritzi-Pauley' specimens and found potassium concentrations of 1400 $mg \cdot kg^{-1}$ in sapwood and from 8000 to 12000 $mg \cdot kg^{-1}$ in false heartwood. This study, however, did not find significant differences in the potassium concentration between both hybrids, both for false heartwood and sapwood.

The calcium concentration practically tripled, from an average value of 908 $mg \cdot kg^{-1}$ in sapwood to 2624 $mg \cdot kg^{-1}$ in false heartwood (Fig. 3). These values coincide with (Janin and Clément 1972), who determined calcium concentrations of 780 $mg \cdot kg^{-1}$ in sapwood and from 1900 to 3500 $mg \cdot kg^{-1}$ in false heartwood. However, (Fukazawa et al. 1985) found a much less marked trend and much lower average concentrations, reaching 44 $mg \cdot kg^{-1}$ in sapwood and 64 $mg \cdot kg^{-1}$ in false heartwood. In addition, this study found that the potassium concentration in false heartwood is greater in the 'NNDV' hybrid than the 'Luisa Avanzo' hybrid (p-value = 0.018), although no differences were found between the sapwood of each hybrid.

Regarding the differences in concentration of other ions of biological importance but not measured in this study, the concentration of the magnesium ion in false heartwood tends to increase systematically by a factor of 4 to 5 (Fukazawa et al. 1985, Janin and Clément 1972). For the sodium ion, (Fukazawa et al. 1985) indicates a reduction in concentration by a factor of 2.4 in false heartwood compared to sapwood. For phosphorous, (Janin and Clément 1972) had determined a reduction in concentration by a factor of 8 to 10 in false heartwood compared to sapwood.

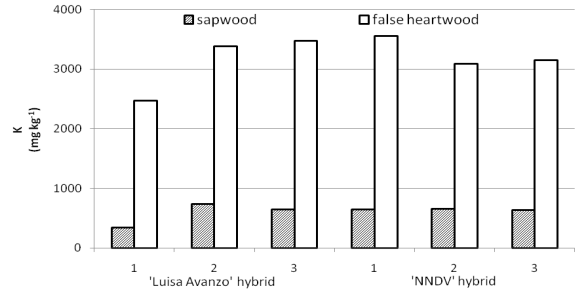


*Fig. 2: Concentration of potassium for false heartwood and sapwood in two Populus hybrids.*

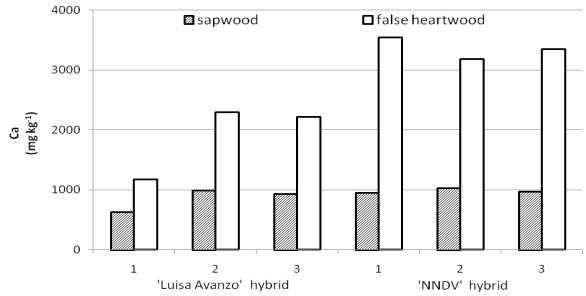


*Fig. 3: Concentration of calcium for false heartwood and sapwood in two Populus hybrids.*

As can be expected, there are differences in the chemical composition that are reflected in the near infrared diffuse reflectance spectra. The principal components analysis (PCA) conducted presented only one discriminating factor (Fig. 4) that results in two clearly distinguishable types of wood. The most relevant spectral ranges for this discrimination (loading matrix from the PCA analysis) are shown in Fig. 5.

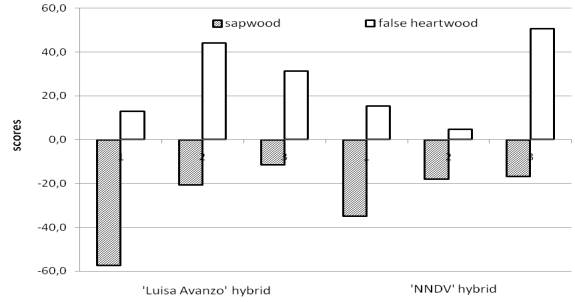


*Fig.4: Principal components analysis for false heartwood and sapwood.*

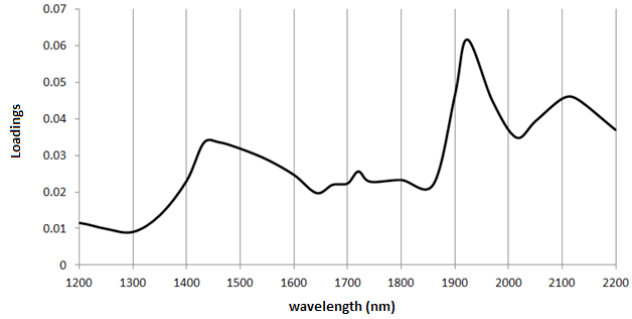


*Fig. 5: Loadings chart for the principal component selected.*

The peaks in diffuse reflectance measured on wood on a wavelength of about 1440 to 1450 nm can be attributed to the first O-H and C-H stretching overtones for lignin and extractives (Schwanninger et al. 2011). The 1850 to 2000 nm range shows the second characteristic

overtone caused by C=O stretching (Workman and Weyer 2007), but this signal is covered by the broad zone of the spectral band generated by the water that presents a maximum at 1942 nm. Subsequently, in the 2000 to 2500 nm range, it becomes difficult to assign functional groups due to the high number of combinations due to couplings between vibrations (Tsuchikawa et al. 2003, Brunner 1996). However, the 2110 nm zone shows combinations of movements specifically in terms of O-H deformations and O-H stretching assigned to cellulose (Ellis and Bath 1940, Schwanninger et al. 2011).

## CONCLUSIONS

The study of the ion concentration in two *Populus* hybrids shows that for the same specimen, the calcium concentration is on average three times higher in the false heartwood zone than in the sapwood zone. Likewise, for the same specimen, the potassium concentration is on average five times higher in false heartwood than in sapwood. The pH increases from slightly acidic in sapwood to slightly basic in false heartwood. The moisture content of wood in freshly cut specimens is notably higher in false heartwood than in sapwood. However, basic density and shrinkage do not differ between the two types of wood. The multivariate statistical method implemented in this study using diffuse reflectance spectroscopy -by examining sawdust with laboratory equipment- was able to discriminate efficiently between sapwood and false heartwood in the *Populus* hybrids studied.

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