Characterization of Cypress Wood for Kraft Pulp Production

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Wood samples of *Cupressus arizonica*, *C. lusitanica*, and *C. sempervirens* were evaluated for chemical, anatomical, and pulp characteristics as raw material for pulp production. Two 17-year-old trees *per* species were harvested, and wood samples were taken at a height of 2 m. Wood chips from *Pinus pinaster* (Portugal) and *P. sylvestris* (Finland) were used as references. *C. arizonica* differed from *C. lusitanica* and *C. sempervirens* with significantly lower ($p < 0.05$) tracheid diameter and wall thickness in the earlywood. The total extractives contents were 3.9%, 3.3%, and 2.5% for *C. lusitanica*, *C. sempervirens*, and *C. arizonica,* respectively, lower than the 5.1% for *P. pinaster* and 4.5% for *P. sylvestris*. Klason lignin content ranged from 33.0 to 35.6%, higher than the 28.0 to 28.7% for the pinewoods. The kraft pulp yields for *C. arizonica*, *C. lusitanica*, and *C. sempervirens* were 37.7%, 36.7%, and 38.7%, respectively, with kappa numbers of 32.0, 31.6, and 28.7, respectively; the yield values were 40.8% and 42.8%, with kappa numbers of 23.4 and 21.0, for *P. pinaster* and *P. sylvestris*, respectively. The cypress species are clearly different from pine in relation to wood pulping behavior. Among the cypress, *C. sempervirens* provided the best pulping results.

Keywords: Cupressus; C. arizonica; C. lusitanica; C. sempervirens; Pulping potential; Extractives; Lignin

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INTRODUCTION

Diversification of tree species is an acknowledged goal for the raw-material supply of forest-based industries that can contribute to global forest sustainability, biodiversity, and adaptation to climatic changes and natural hazards. This applies strongly to the pulp and paper sector, for which the environmental problems resulting from increasing world consumption of paper have stimulated research into the use of alternative raw materials in response to environmental and economic pressures.

The use of cypress species as a pulping raw material may be envisaged in this context as an alternative to conventional long-fiber softwoods such as *Pinus* and *Picea* spp. Cypress are conifer evergreen trees or shrubs native to the warm temperate areas of the Northern hemisphere. The genus comprises about 15 species distributed throughout the western United States, Mexico, northern Central America, the Mediterranean region, north Africa, and from southern Asia to Japan (Laubenfels 2009). In Europe, they are primarily distributed within the southern countries.

There is not much information on the wood characterization of cypress species. The studies on cypress wood properties include chemical composition (Okino *et al.* 2010), physical and mechanical properties (Hashemi and Kord 2011; Kothiyal *et al.* 1998), acoustic properties (Roohnia *et al.* 2011), decay resistance and properties of oriented strand board (Okino *et al.* 2009; 2008), particleboards, flakeboards, and cement-bonded boards (Okino *et al.* 2005) and pulp (Guha *et al.* 1969, 1971).

The wood characteristics, namely fiber length, width and wall thickness, of 27-year-old *Cupressus macrocarpa* were only slightly inferior to those of *Pinus radiata*, and its lower coarseness (0.138 $mg.m^{-1}$) with a basic density of 0.405 $g.cm^{-3}$ was found to be ideal for incorporation with other softwoods to produce writing and printing papers (Somerville 1993). The pulping potential of wood samples from *C. arizonica*, *C. lusitanica*, and *C. sempervirens* was investigated in a preliminary study by the present team of researchers (Silva *et al.* 2007; Esteves *et al.* 2004; Almeida *et al.* 2003), and the paper characteristics were evaluated (Anjos *et al.* 2014). The results showed an acceptable pulp yield and interesting fiber characteristics.

In this paper, we study the wood quality for pulp production of three cypress species that grow in Portugal in an experimental stand located in Castelo Branco (*C. arizonica*, *C. lusitanica*, and *C. sempervirens* (var. *horizontalis*)) by investigating their anatomical features, chemical composition, and kraft pulping aptitude. The underlying rationale is that *Cupressus* species can be used to diversify the forest in the context of species enrichment and decrease of fire risk, while cypress wood can be used as long-fiber reinforcement in eucalypt pulps for printing papers in addition to currently used pine pulps (*e.g.*, *P. pinaster* and *P. sylvestris*).

EXPERIMENTAL

Sampling

The cypress trees were harvested in a mixed 17-year-old cypress stand in a recreation area (39° 49' N, 7° 30' W) with low and variable stand densities. The soil was a Regosol (WRB, ZN6), with a pH of around 5.7 and organic matter content of around 3.1%. The stand has an average rainfall of 731 mm/year, air relative humidity of 72.5% at 9 a.m., mean annual hours of sunshine of 2,880 h, and mean temperature between 8.1 and 24.9 ºC.

Two trees each of *C. arizonica*, *C. lusitanica*, and *C. sempervirens* were randomly chosen in the stand, and total height and tree diameter at breast height were measured (Table 1).

Table 1. Tree Diameter at 1.3 M (DBH) and Total Height of the Sampled Cypress Trees

	DBH (cm)		Height (m)	
	Tree 1	Tree 2	Tree 1	Tree 2
C. arizonica	19.0	17.0	7.9	7.9
C. lusitanica	13.0	11.5	5.8	4.6
C. sempervirens	13.5	9.0	7.8	6.0

The trees were harvested, and disc samples of about 10-cm thickness were cut at 2 m of stem height above the ground. Representative commercial wood chips from *P. pinaster* (from tree stems with approximately 25 years of age) grown in Portugal and from *P. sylvestris* grown in Finland were used as references. The wood discs were subdivided transversely into thinner discs for density determination, anatomical characterization, chemical analysis, and pulping.

Density was determined on the disc as basic density using water displacement for volume determination according to TAPPI 258 om-89 (1989).

Wood Morphological Characteristics

The wood morphological characteristics were determined along one randomly selected radius at four distances from the pith: 5, 25, 50, and 85% of the total radius.

The wood samples were cut with 5x5 mm^2 in cross section (tangential and radial directions) and 15 mm in length (longitudinal direction). The samples were boiled to remove the air during approximately 2 h with Mili-Q water and softened in a 1:10 solution of glycerin and water for several days.

The cross sections of the wood samples were first planed with a still knife to prepare them for microscopic examination. With a sliding microtome, thin sections of 18 μm thickness were taken for microscopic analysis, using well-sharpened knifes with type C sections. The knife was angled in a way that at least one quarter of the blade was used when taking a section. The samples were suspended with sodium hypochlorite solution, washed with water and colored with fuchsia for cell wall enhancement and better measurements. The tangential tracheid diameter and wall thickness were determined in latewood and earlywood of the annual ring by measuring 100 cells at each radial sampling point.

The wood images were acquired with a reflective light microscope (Leica, DMLL, Germany) at a magnification of 200 X and analyzed with a Qwim 500® image analysis system (Leica, England).

The cypress fiber images were acquired with an Hitachi S-2700 scanning electron microscope (SEM) operating at an accelerating voltage of 20 kV. The sample material was fixed in a 2.5% glutaraldehyde solution overnight at 4 °C, dehydrated in a graded ethanol series, dried with liquid CO_2 at critical point, and subsequently coated with gold by cathodic spraying.

Chemical Analysis

The wood discs of cypress species were manually chipped to approximate dimensions of 0.5 cm x 1 cm x 2 cm (radial, tangential, and axial). A representative sample was milled in a hammer mill and sieved, and the 40- to 60-mesh fraction was used for chemical analysis according Anjos *et al.* (2013).

The wood meal samples were extracted successively with dichloromethane, ethanol, and water in a Soxtec System HT 1043 Extraction Unit apparatus (Sweden), according to TAPPI T204 cm-07 (2007). In this method, 2 g of the dried sample was extracted using 40 mL of dichloromethane at 110 ºC for 1 h, followed successively with absolute ethanol, then water, at 160 and 200 ºC, respectively. All analyses were conducted on quadruplicate aliquots, and the results are reported as a percentage of the original oven-dry sample mass.

Klason and acid-soluble lignin contents were determined in the extracted material according to TAPPI T222 om-02 and TAPPI T250 um-85. The extractive-free wood (0.333 g) was pre-hydrolyzed with sulfuric acid (72%, 5.0 mL) at 20 ºC for 2 h, followed by dilution and autoclaving for 1 h at 120 ºC. Klason lignin was determined as the mass of the solid residue after drying at 103 ± 2 ºC. The acid-soluble lignin was determined on the filtrate by measuring the absorbance at 205 nm. Total lignin content was calculated as the sum of Klason lignin and acid-soluble lignin. All analyses were conducted on duplicate aliquots.

Pulp

The wood chips were pulped in a forced circulation reactor under the following conditions (two replicate cookings per species); the wood chip charge was 1000 g oven dry (o.d.) wood, and the kraft cooking conditions were as follows: active alkali 25% (NaOH), sulfidity 30%, liquor/wood ratio 5:1, pulping temperature 170 ºC, time to temperature 90 min, and time at temperature 150 min. The pulped material was washed and screened on an L&W screen with 0.3 mm slot width. The screened pulp yield and rejects, kappa number (KN) (ISO 302 2004), and pulp viscosity (ISO 5351/1 2010) were determined according to standard methods. Residual alkali was determined in the black liquor by acid titration to pH 10.5, after dilution and barium chloride addition. The effective alkali consumption was estimated considering the initial 21.25% effective alkali charge.

Data Analysis

A one-way ANOVA was performed to determine significant differences among species with a Scheffé post-hoc test (95% confidence level). The results were also subjected to a multivariate analysis (principal component analysis). All experimental data were analyzed using StatSoft Statistics® (version 7).

RESULTS AND DISCUSSION

Morphological Characteristics

Tracheid diameter and wall thickness are important anatomical characteristics related to wood density, pulp properties, and paper characteristics (Kibblewhite and McKenzie 1999; Zobel and Buijtenen 1989). Table 2 summarizes the mean tracheid dimensional features in earlywood and latewood for the three cypress species.

The tracheid diameters for the pines were significantly higher than those observed for the cypress species as observed in Fig 1. The cell wall thickness of pines is lower than that of *C. sempervirens* and *C. lusitanica* but similar to the value observed for *C. arizonica*.

The tracheid diameter and wall thickness in the earlywood of *C. arizonica* were significantly lower ($p < 0.05$) than those observed in *C. lusitanica* and *C. sempervirens*. Only latewood tracheid tangential diameter did not differ significantly for the three cypress species. This is also supported by principal component analysis (which explains 98% of the total variance) that clearly separated *C. arizonica* from *C. lusitanica* and *C. sempervirens* regarding the morphological characteristics measured.

The wood of the three cypress species was rather homogeneous regarding earlywood and latewood, with very similar tracheid diameter, although the wall thickness was higher in the latewood (Table 2).

The dissociated tracheids of the cypress species showed different transversal sizes compared to the pine tracheids, as shown in Fig. 1.

Table 2. Mean Tracheid Transverse Dimensions in Earlywood and Latewood of 17-Year-Old Trees of *C. arizonica*, *C. lusitanica*, and *C. sempervirens*

	Earlywood		Latewood	
	Tangential diameter (µm)	Cell wall thickness (µm)	Tangential diameter (µm)	Cell wall thickness (µm)
C. arizonica	20.2 ± 1.2 ^{a}	2.6 ± 0.2 ^{a}	19.8 ± 1.1 ^{a}	3.8 ± 0.2 ^{a}
C. lusitanica	22.5 ± 2.0 ^{a,b}	3.0 ± 0.4 ^{a,b}	21.6 ± 1.2 ^{a}	4.1 ± 0.5 ^{b}
C. sempervirens	23.2 ± 1.5 ^{b}	3.4 ± 0.4 ^{b}	21.7 ± 2.0 ^{a}	4.4 ± 0.4 ^{b}
P. pinaster	26.2 ± 1.3 ^{c}	2.2 ± 0.3 ^{c,a}	24.2 ± 1.3 ^{b}	3.5 ± 0.3 ^{a}
P. sylvestris	26.4 ± 1.4 ^{c}	1.9 ± 0.2 ^{c}	24.6 ± 1.4 ^{b}	3.6 ± 0.2 ^{a}
Means with the same letter in the same columns do not differ significantly ($p < 0.05$)				

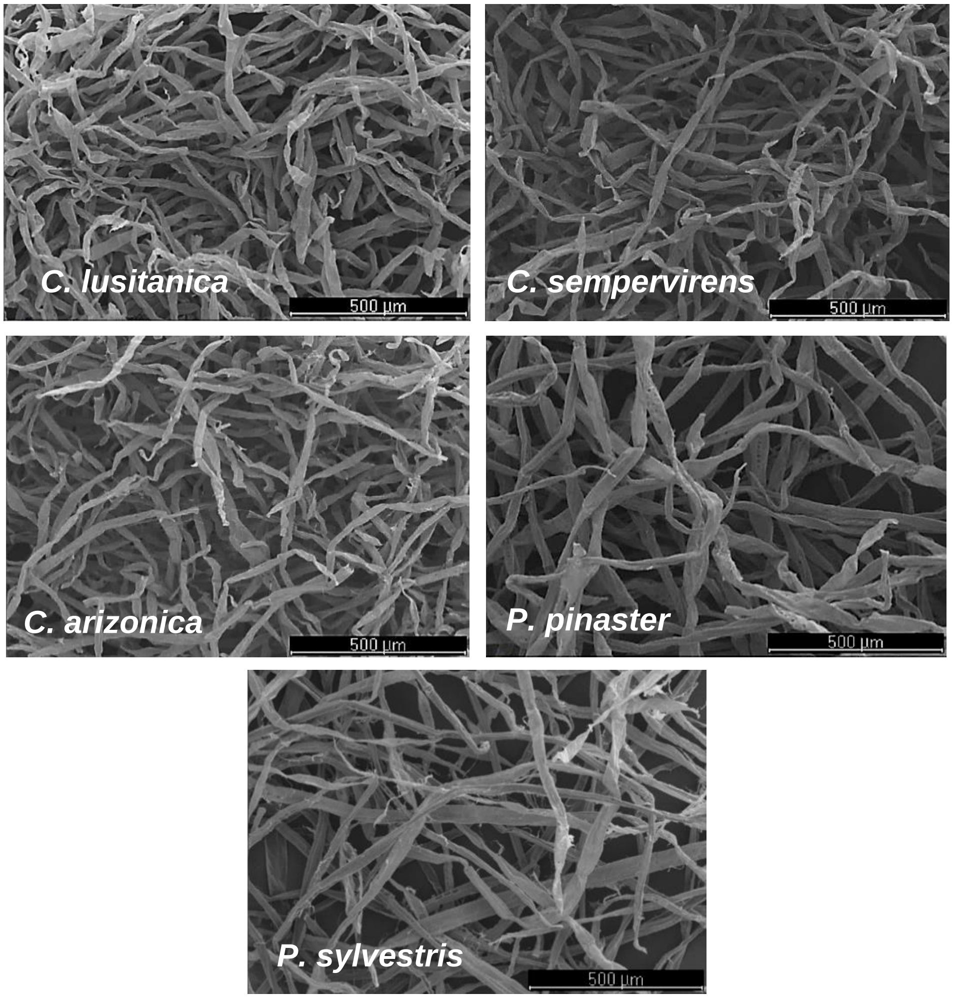

Fig. 1. Scanning electron micrographs of the dissociated tracheids of *C. lusitanica*, *C. sempervirens*, *C. arizonica*, *Pinus pinaster*, and *Pinus sylvestris*

The radial variation of the tracheid dimensions is shown in Figs. 2 and 3. There was a small increase in the wall thickness of earlywood tracheids in the three species and a decrease in the earlywood tracheid diameter for *C. lusitanica* and *C. sempervirens*.

However, the differences between radial positions (*e.g.*, resulting from differences in cambial age) were small in magnitude.

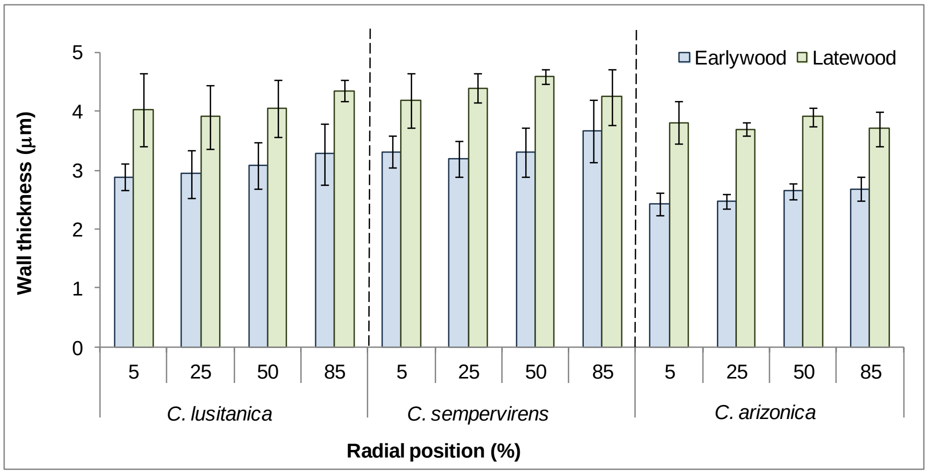

Fig. 2. Wall thickness in latewood and earlywood of *C. arizonica*, *C. lusitanica, and C. sempervirens*

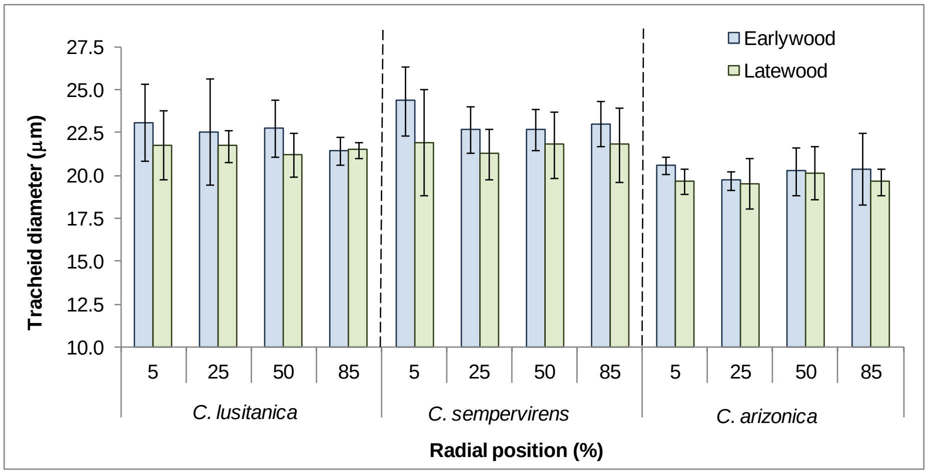

Fig. 3. Tracheid diameter in latewood and earlywood of *C. arizonica*, *C. lusitanica,* and *C. sempervirens*

Wood Density

The wood density is provided in Table 3 for the three cypress species and compared to that of the two pine species.

Table 3. Wood Basic Density for *C. arizonica*, *C. lusitanica*, *C. sempervirens*, *P. pinaster*, and *P. sylvestris* (mean and standard deviation of 4 samples)

	Density (g.cm^{-3})
C. arizonica	0.430 ± 0.10 $^{\mathbf{a}}$
C. lusitanica	0.432 ± 0.13 $^{\mathbf{a,b}}$
C. sempervirens	0.453 ± 0.15 $^{\mathbf{b,c}}$
P. pinaster	0.465 ± 0. 20 $^{\mathbf{c}}$
P. sylvestris	0.423 ± 0.15 $^{\mathbf{a}}$
Means with the same letter do not differ significantly ($p < 0.05$)	

The wood density of *C. sempervirens* did not differ significantly from that of *P. pinaster*, while that of *C. arizonica* and *C. lusitanica* did not differ from *P. sylvestris*. The wood basic density of *C. sempervirens* was similar to the values reported by Hashemi and Kord (2011) and Paraskevopoulou (1991), and that of *C. lusitanica* agrees with the values reported by Elzaki and Khider (2013).

Chemical Composition

The chemical composition (% by mass on o.d. wood) of the three cypress species and the pine references is reported in Tables 4 and 5.

Table 4. Extractives Content of *C. arizonica*, *C. lusitanica*, *C. sempervirens P. pinaster*, and *P. sylvestris*

	Dichloromethane	Ethanol	Water	Total
C. arizonica	0.29 ± 0.08 ^{a}	1.28 ± 0.20 ^{b}	0.89 ± 0.12 ^{a,b}	2.46 ± 0.30 ^{a}
C. lusitanica	0.83 ± 0.18 ^{c}	2.27 ± 0.40 ^{d}	0.77 ± 0.30 ^{a}	3.87 ± 0.70 ^{c}
C. sempervirens	0.34 ± 0.12 ^{b}	2.00 ± 0.66 ^{c}	1.00 ± 0.27 ^{b,c}	3.34 ± 0.66 ^{b}
P. pinaster	2.85 ± 0.04 ^{e}	1.05 ± 0.07 ^{a}	1.15 ± 0.23 ^{c}	5.05 ± 0.17 ^{e}
P. sylvestris	2.45 ± 0.04 ^{d}	1.15 ± 0.08 ^{a,b}	0.87 ± 0.11 ^{a}	4.47 ± 0.19 ^{d}
Means with the same letter in the same columns do not differ significantly ($p < 0.05$)				

Table 5. Lignin Content of *C. arizonica*, *C. lusitanica*, *C. sempervirens* (Mean of Two Trees), *P. pinaster*, and *P. sylvestris*

	Klason	Soluble	Total
C. arizonica	35.62 ± 1.72 ^{c}	0.46 ± 0.22 ^{a}	36.08 ± 1.79 ^{b}
C. lusitanica	34.13 ± 4.12 ^{b,c}	0.56 ± 0.04 ^{a}	34.69 ± 4.12 ^{b}
C. sempervirens	32.96 ± 1.05 ^{b}	0.54 ± 0.08 ^{a}	33.50 ± 1.07 ^{b}
P. pinaster	28.70 ± 0.63 ^{a}	0.42 ± 0.10 ^{a}	29.12 ± 0.53 ^{a}
P. sylvestris	28.04 ± 0.96 ^{a}	0.44 ± 0.10 ^{a}	28.48 ± 1.05 ^{a}
Means with the same letter in the same columns do not differ significantly ($p < 0.05$)			

The cypress species exhibited lower extractives contents than the pine species (Table 4). Most differences were related to the content in non-polar extractives soluble in dichloromethane (including *e.g.* fats, waxes, terpenoids, and higher aliphatic alcohols), which was significantly higher in the pines. Even if the differences between the three cypress species were comparatively small, they had statistical significance ($p < 0.05$). The cypress wood lignin content (Table 5) was significantly higher than that of the pines, with averages of 34% and 29% in cypress and pine woods, respectively. Considering the combined effect of extractives and lignin content, it can be inferred that the carbohydrate content of the cypress samples was 3 to 5.5 percent values lower than the corresponding values for the pines.

There is very little data on the chemical composition of cypress wood. For *C. glauca*, Okino *et al.* (2008) found 32% Klason lignin and 1.1% extractives (with toluene–ethanol 2:1, ethanol and hot water), while Hafizoglu and Usta (2005) report similar results for Klason lignin in *C. sempervirens*.

In summary, the comparison of the chemical composition of the cypress and pine woods suggests that the pulping potential of the cypress wood samples tested in the present study is lower than that of commercial pine pulp wood.

Pulping Parameters

The results for the kraft pulping are represented in Table 6. The pulp yield from the cypress woods is lower than that of the pines (37.7% *vs.* 41.8%), and the pulps attained a lower delignification degree, as determined by the higher kappa number (31 *vs.* 22). The differences in pulp yield would be even more evident if the comparison were made at the same pulp kappa number.

These differences are in accordance with the differences in lignin content of cypress and pine woods. As a consequence, the alkali consumption in the pulping of the cypress wood samples was higher than for the pine samples (Table 6). Of the three cypress species, *C. sempervirens* showed the best pulping results, *i.e.* higher pulping yield, lower kappa number (higher delignification), and lower alkali consumption, in accordance with its lower lignin content.

The pulp yield obtained with *P. pinaster* wood was less than that obtained with *P. sylvestris* following its higher extractive content and slightly higher lignin content, in accordance with values reported by Esteves *et al.* (2005). The differences reported in Table 6 would be even slightly higher if a reference kappa number of *e.g.* 30 was considered.

It should be noted that the studied cypress trees were relatively young and that the use of older raw materials should improve the pulp yield (Molteberg and Hoibo 2006).

Table 6. Pulping Results of *C. arizonica*, *C. lusitanica*, *C. sempervirens*, *P. pinaster* and *P. sylvestris*

	Pulp Yield (%)	Kappa number	Viscosity ($cm^3.g^{-1}$)	Rejects (%)	Effective Alkali Consumption (%)
C. arizonica	37.7 ± 0.6 ^{a,b}	32.0 ± 1.7 ^{c}	657 ± 21 ^{a}	0.7 ± 0.4 ^{a}	17.4 ± 0.3 ^{b}
C. lusitanica	36.7 ± 2.8 ^{a}	31.6 ± 3.4 ^{c}	772 ± 25 ^{c}	0.9 ± 0.6 ^{a}	18.0 ± 0.3 ^{c}
C. sempervirens	38.7 ± 1.2 ^{b}	28.7 ± 1.8 ^{b}	717 ± 34 ^{b}	0.6 ± 0.4 ^{a}	17.3 ± 0.4 ^{a,b}
P. pinaster	40.8 ± 0.9 ^{c}	23.4 ± 2.0 ^{a}	830 ± 22 ^{d}	0.0 ± 0.0	17.0 ± 0.5 ^{a,b}
P. sylvestris	42.8 ± 0.4 ^{c}	21.4 ± 0.9 ^{a}	890 ± 38 ^{e}	0.0 ± 0.0	16.9 ± 0.3 ^{a}
Means with the same letter in the same columns do not differ significantly ($p < 0.05$)					

Regarding pulp intrinsic viscosity, the results for cypress wood species were lower. This can be partially attributed to wood age, according to Goswami *et al.* (1996).

A principal component analysis was performed with the wood chemical data and pulping results from the three cypress and two pine species. Two factors were found to represent 84.2% of total variance.

Figure 4 represents the projection cases onto the system of vectors generated by using all studied parameters, showing how the values of the different woods are correlated. The pines have clearly different characteristics from the cypress species, as shown by the distribution along Factor 1. Factor 2 indicates that *C. lusitanica* presents a different pulping performance than *C. sempervirens* and *C. arizonica*.

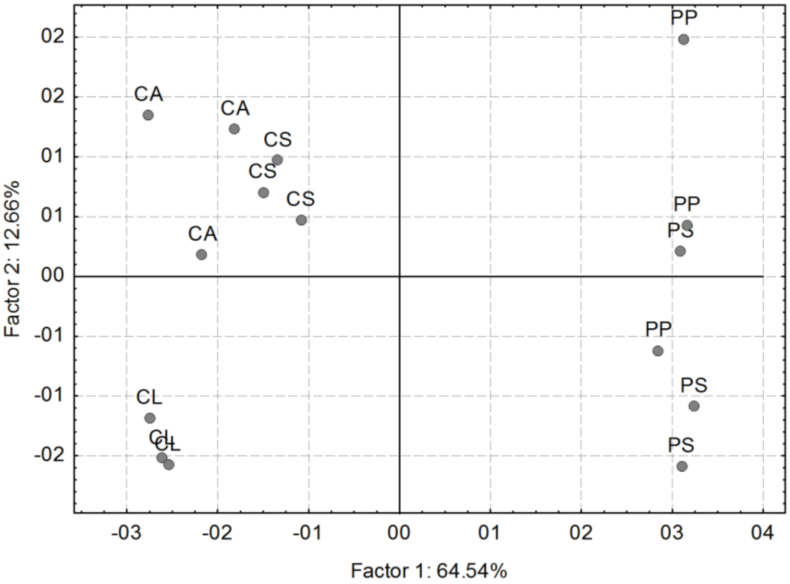

Fig. 4. Projection diagram of the cases on the factor plane (Factor 1 *vs.* Factor 2): CA, *C. arizonica*; CL, *C. lusitanica*; CS, *C. sempervirens*; PP, *P. pinaster*; PS, *P. sylvestris*

CONCLUSIONS

1. The cypress species *C. arizonica*, *C. lusitanica*, and *C. sempervirens* may be considered as potential raw materials for pulping, although the pulping results are significantly inferior to those obtained with commercial pulpwood pine species (*P. pinaster* and *P. sylvestris*).
2. The three cypress species differ from each other, as well as from the pine species, with respect to their chemical and pulping characteristics.
3. The wood from *C. sempervirens* appears to be better suited for pulping than the other cypress species, with lower Klason lignin, higher pulp yield, and better delignification, than the other cypress species.

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