

Variation of the chemical composition of Pyrenean oak (*Quercus pyrenaica* Willd.) heartwood among different sites and its relationship with the soil chemical characteristics

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Abstract This work presents the results of a study on the variation of the chemical composition of Pyrenean oak (*Quercus pyrenaica* Willd.) heartwood among different sites and its relationship with the soil chemical characteristics. The chemical characteristics of the heartwood are quantified in terms of proximate and ultimate analysis, calorific value and ash composition. Subsequently, the relationship between the wood chemical characteristics and the soil characteristics was also examined. The results showed no significant differences between the wood chemical characteristics from different sites. There are, however, significant differences between trees from the same site regarding the wood ash content, the calorific value and the contents of nitrogen and hydrogen. Strong correlations were observed between the heartwood ash contents of BaO, MnO, CuO and TiO₂ and the soil nitrogen, phosphorous and potassium and the soil exchangeable base contents.

Keywords *Quercus pyrenaica* Willd. · Wood chemical composition · Soil characteristics

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Introduction

The study of the wood chemical composition is critical for obtaining pertinent information about its characteristics and to enable better use of wood resources (e.g., Fengel and Wegener 1989; Sjöstrom 1993; Rowell 2005). There are no detailed studies in the literature on the chemical characteristics of Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood, and there does not exist any study on the relationship between its chemical composition and the soil chemical characteristics. This is an important issue for the species knowledge (Santos et al. 2012) and for the forest management (Johnson 1993; Sjöstrom 1993). Oak forests of these species are very important because of their multiple functions (ecological, economic and social) by the goods and services they provide. The wood from these species may provide a manifold of potential uses, such as flooring, carpentry, veneer, cooperage and energy production (Carvalho 2012; Santos et al. 2012). The sawtimber discards the sapwood for most of these uses because of its low strength and vulnerability to insect attack.

The knowledge of the chemical characteristics of the wood, including its ash composition, may be important for various uses or fields (Hillis 1984; Tsoumis 1991; Quaak et al. 1999; Mandre 2006). For example, the allocation of woody biomass for heating is directly related to its chemical composition in a way that allows evaluating biomass quality for burning by analyzing the wood chips' content of each chemical element (Nordin 1994; Demirbas 2004; Bech et al. 2009; Huang et al. 2009). The chemical composition, determined by the relative proportion and interaction of the different chemical components in their structure (Rowell 2005), is also related to the durability of the wood, color, mechanical strength, hygroscopicity and heating value, among other physical and mechanical

characteristics. The ash generated as a by-product of combustion can be used as soil fertilizer (Naylor and Schmidt 1986; Demeyer et al. 2001; Pitman 2006). Application of ash on the ground for recycling nutrients such as potassium, calcium, magnesium and phosphorus can be made for amendment or balancing of the nutrients of agricultural and forest soils (Naylor and Schmidt 1986; Campbell 1990; Steenari et al. 1999; Reijnders 2005).

According to several authors (e.g., Pettersen 1984; Shmulsky and Jones 2011), it is also interesting to know the variability of the wood chemical characteristics that can be attributed to factors such as the species, genetic characteristics and environmental conditions of the tree growth. The study of the wood chemical composition and its relationship with the soil characteristics is also relevant to evaluate the nutrient recycling in the forest (Ranger and Bonneau 1984; Khanna and Fabiao 2002). Information about the quantity of nutrient exports resulting from logging and the availability of nutrients in the forest soils is essential topics in sustainable forestry (Nykqvist 1976; Olsson et al. 1996; Brandtberg and Olsson 2012). Moreover, the intensity of forest biomass exploitation for some species affects the soil fertility and its relationship with the nutrients available in the soil (Watmough 2014).

The aim of the present work is to evaluate the variation of the chemical composition of Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood among different sites and its relationship with the soil chemical characteristics.

Materials and methods

The wood chips used in this study were obtained from Pyrenean-oak stands from the north of Portugal, in four different sites, having two different types of parent material (schist and granite) and soil type (Table 1). The sites present a mean annual precipitation ranging from 1080 to 1290 mm and a mean annual temperature ranging from 9.7 to 11.0 °C. The vegetation was a pure stand dominated by Pyrenean-oak with the minor presence of other tree species, namely common cherry (*Prunus avium* L.), madrone (*Arbutus unedo* L.) and Plymouth pear (*Pyrus cordata* Desv.).

In each site, a plot of 500 m² with homogeneous size classes was established and two dominant trees were selected and harvested for the present study based on the homogeneity of the stand and the indications of Tennent and Burkhart (1981). For the purposes of this study, all the chemical analyses were carried out using heartwood. From each tree and logs, the central board was sawed. Wood slats of the heartwood were obtained with approximately 1 cm thickness, which were subsequently cut into cubes of approximately 1-cm edges and then ground in a mill for chemical analysis. At each site, a soil profile was dug and the horizons, properties and materials were observed for a preliminary soil classification. The final classification was made after the analytical evaluation of the physical and chemical characteristics of the soil (Van Reeuwijk 2002) from the Laboratory of Soil. The classification of the soil type was based on the FAO classification system (FAO 2015). Soil types and textures in Table 1 are the most common found in Pyrenean-oak forests (Carvalho and Parresol 2004; Carvalho 2012). At each site, a composite soil sample was obtained from each horizon and stored in plastic bags. Soil sampling was made at all soil profiles and depths. The chemical parameters analyzed were the following: soil reaction (pH(H₂O)), organic matter (OM), nitrogen (N), phosphorus pentoxide (P₂O₅), potassium oxide (K₂O), calcium (Ca), magnesium (Mg) potassium (K), sodium (Na), the sum of exchangeable bases (SEB), exchangeable acidity (AE) and the degree of base saturation (DBS) (Table 2). The soil composition was different at the four sites, mainly in extractable and exchangeable cationic bases, with the Macedo site showing higher amounts of these elements.

The chemical characteristics of the heartwood were evaluated in terms of proximate analysis, ultimate analysis, calorific value and composition of the ashes. The proximate and ultimate analysis and the calorific values of the heartwood were carried out following the procedures specified in the standards ASTM-E-870, EN 14918 and EN 14775, and the chemical composition of the heartwood ash was determined using X-ray fluorescence spectroscopy.

The heartwood and soil chemical parameters were analyzed to determine whether there were significant

Table 1 Physical characteristics of the studied sites

Site	Location lat., long.	Stand age (years)	Parent material	Soil type	Soil depth (cm)	Soil texture
1-Macedo	41°37'N, 6°58'W	53	Schist	<i>Cambisol</i> (CM)	95	Silty loam
2-Bragança	41°51'N, 6°46'W	52	Schist	<i>Luvisol</i> (LV)	150	Loam
3-Chaves	41°45'N, 7°18'W	52	Granite	<i>Cambisol</i> (CM)	140	Sandy loam
4-V.P. Aguiar	41°27'N, 7°40'W	55	Granite	<i>Leptosol</i> (LP)	45	Sandy

Table 2 Chemical analysis of the soils

Site	Macedo	Bragança	Chaves	V.P. Aguiar
pH (H ₂ O)	5.5	5.0	4.9	4.9
OM (%)	1.5	0.4	7.4	4.1
N (%)	0.095	0.032	0.750	0.197
P ₂ O ₅ (mg g ⁻¹)	8.2	3.9	11.7	6.0
K ₂ O (mg g ⁻¹)	189	84	65	108
Ca (mg g ⁻¹)	2.72	0.45	0.47	1.18
Mg (mg g ⁻¹)	2.40	0.60	0.07	0.68
K (mg g ⁻¹)	0.27	0.13	0.08	0.21
Na (mg g ⁻¹)	0.29	0.10	0.11	0.08
SEB (mg g ⁻¹)	5.68	1.28	0.73	2.15
AE	0.88	2.97	2.19	1.42
DBS (%)	84.1	34.9	25.7	60.2

variations between trees within the same site and different sites (intra- and interpopulation variation). An analysis of variance was performed for the heartwood elements in order to examine the significance of variance from trees and sites. Further analysis was undertaken to evaluate the relationship between the heartwood and the soil chemical compositions from different sites. A principal component analysis (PCA) with standardized data allowed for an enhanced understanding of the data structure, the interactions between variables and a better interpretation of the results. The correlation matrix was analyzed with the aid of the Bartlett test of sphericity (Gauch 1982; Jolliffe 2002) in order to evaluate the occurrence of significant correlations among the variables. A correlation analysis between the heartwood and the soil chemical elements was performed through the Pearson correlation coefficient (Steel and Torrie 1997; Norman and Streiner 2007).

Results

Table 3 shows the results of the proximate and ultimate analysis, calorific value and ash composition for each sample. The moisture and volatile matter contents are very similar among the samples, but both the ash and the fixed carbon contents show high variability. The average values of the moisture, volatile content, ash and fixed carbon are 11, 86, 0.2 and 3 wt%, respectively, while the average values of the LCV (lower calorific value) and HCV (higher calorific value) are 16.79 and 18.14 MJ kg⁻¹, respectively. The ultimate analysis shows average values of 49 wt% for C, 44 wt% for O, 0.05 wt% for N, 6 wt% for H and 0.01 wt% for S.

As for the composition of the heartwood ash, the most common oxides are CaO with 37 wt%, K₂O with 31 wt%, SO₃ with 11 wt%, MgO and P₂O₅ both with 4 wt% and

Al₂O₃ with 2 wt%. The remaining oxides in the heartwood ash present contents lower than 2 wt% (Table 3).

The analysis of variance showed no significant differences between sites in the amounts of the chemical elements of the heartwood, but significant differences between trees in the same site were observed for some variables, namely for the ash content, both for the LCV and the HCV, and for the contents of N and H (Table 4).

In the case of the ash composition, the analysis of the variance did not reveal significant differences between sites and trees in the same site (Table 5).

The PCA for the heartwood chemical characteristics showed that the first four components explained 89.4% of the observed variance and the first two components 57.5%. Figure 1 shows components 1 and 2 from the PCA for the chemical variables of the Pyrenean-oak heartwood. The first principal component correlates positively with LCV, HCV, H, Al₂O₃, BaO, CaO, Cr₂O₃, Na₂O, SiO₂ and SrO. The second principal component shows positive correlations with N, CuO, Fe₂O₃, TiO₂, ZnO and SO₃. The third principal component relates positively with BaO, MnO and Rb₂O.

Figure 2 shows components 1 and 2 from the PCA for the chemical variables of the soil and of the Pyrenean-oak heartwood, which explains 84.1% of the total variance. The first principal component relates positively with some heartwood ash elements such as BaO, MnO, TiO₂, ZnO, SO₂, Fe₂O₃ and CuO, and with the soil OM and N. This component relates negatively with most of the soil chemical characteristics (K₂O, Ca, Mg, K, Ca, SEB, DBS, pH). The second component correlates positively with LCV, HCV and O.

Table 6 shows the significant correlations between the chemical variables of the soil and of the Pyrenean-oak heartwood.

Several soil chemical characteristics (K₂O, Ca, Mg, K, SEB, EA, DBS and pH) present significant negative correlations with the heartwood ash contents of BaO and MnO. The soil N correlates positively with CuO and TiO₂, and the soil P₂O₅ with CuO.

Discussion

The results showed that for a large number of heartwood chemical parameters there are no significant differences between sites. Despite the differences between soils, with the exchangeable bases of the soil of site 1 (Macedo) being higher than those of the other sites, the heartwood chemical composition is similar for all sites. For some elements, however, there are significant differences between trees located in the same site, namely regarding the ash content, both the LCV and the HCV and both the N and the H contents. This significant variation in the heartwood

Table 3 Chemical analysis of the Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood samples from the various sites

Site sample	1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2	Mean	CV (%)
<i>Proximate analysis (wt%)</i>										
Moisture	9.7	10.9	11.5	11.7	10.2	10.8	11.4	10.8	10.88	6.2
Ash	0.2	0.1	0.4	0.2	0.2	0.1	0.4	0.2	0.23	51.8
Volatile matter	85.3	84.8	86	86	85.3	87	86.2	83.4	85.50	1.3
Fixed carbon	4.8	4.2	2.1	2.1	4.3	2.1	2.0	5.6	3.40	43.5
Total	100	100	100	100	100	100	100	100		
LCV (MJ kg ⁻¹)	16.70	18.30	15.20	18.10	16.70	17.80	14.70	16.80	16.79	7.8
HCV (MJ kg ⁻¹)	18.00	19.70	16.50	19.40	18.10	19.20	16.00	18.20	18.14	7.3
<i>Ultimate analysis (wt%, dry basis)</i>										
Carbon (C)	45.9	49.1	53.2	48.9	46.6	49.2	52.8	49.8	49.44	5.2
Oxygen (dif.) (O)	47.69	44.37	40.05	44.49	46.90	44.26	40.45	43.52	43.96	5.9
Nitrogen (N)	0.1	0.02	0.04	0.005	0.1	0.005	0.08	0.03	0.05	84.6
Hydrogen (H)	6.1	6.4	6.29	6.4	6.19	6.43	6.25	6.43	6.31	2.0
Sulfur (S)	0.011	0.01	0.02	0.01	0.012	0.01	0.02	0.02	0.01	34.8
Ash	0.2	0.1	0.4	0.2	0.2	0.1	0.4	0.2	0.23	51.8
Total	100	100	100	100	100	100	100	100		
C/N	459	2455	1330	9780	466	9840	660	1660	3331	121.7
C/H	7.52	7.67	8.46	7.64	7.53	7.65	8.45	7.74	7.83	5.0
<i>Composition of ash (wt%, dry basis)</i>										
Al ₂ O ₃	0.21	2.85	0.22	1.30	0.48	7.12	0.44	3.72	2.04	119.2
BaO	0.22	0.64	0.56	0.85	1.10	0.80	0.36	0.76	0.66	42.5
CaO	24.80	48.10	20.70	44.40	36.70	34.70	32.2	55.40	37.13	31.5
Cl	0.03	0.12	0.72	0.29	0.15	0.11	0.58	0.07	0.26	99.2
Cr ₂ O ₃	0.001	0.02	0.02	0.02	0.001	0.07	0.008	0.04	0.02	99.3
CuO	0.09	0.12	0.07	0.12	0.44	0.23	0.04	0.11	0.15	84.9
Fe ₂ O ₃	0.12	0.76	0.17	0.22	7.10	1.20	0.2	0.27	1.25	190.7
K ₂ O	28.60	26.40	60.20	32.40	26.90	21.00	51.3	5.19	31.50	54.7
MgO	1.60	4.46	4.72	5.05	0.87	3.67	3.66	10.50	4.32	67.3
MnO	0.04	0.20	1.25	3.11	3.10	2.66	0.33	0.71	1.43	93.0
Na ₂ O	0.22	2.20	0.86	0.72	0.99	3.50	0.75	1.40	1.33	79.1
NiO	0.02	0.04	0.04	0.02	0.01	0.01	0.007	0.05	0.02	64.7
P ₂ O ₅	0.94	3.42	5.19	2.30	2.20	4.51	3.65	13.20	4.43	85.7
Rb ₂ O	0.05	0.04	0.11	0.09	0.13	0.05	0.2	0.01	0.09	71.6
SiO ₂	1.80	4.48	1.00	2.41	2.60	12.00	1.68	2.80	3.60	98.6
SO ₃	40.90	5.57	2.63	6.28	16.20	7.83	3.09	5.14	10.96	117.0
SrO	0.22	0.38	0.26	0.38	0.59	0.41	0.32	0.55	0.39	33.2
TiO ₂	0.04	0.09	0.10	0.03	0.33	0.20	0.15	0.03	0.12	84.5
ZnO	0.01	0.01	0.02	0.01	0.09	0.03	0.04	0.04	0.03	84.3
Total	100	100	100	100	100	100	100	100		

wt weight, *LCV* lower calorific value, *HCV* higher calorific value, *C/N* carbon/nitrogen ratio, *C/H* carbon/hydrogen ratio, *Al₂O₃* aluminum oxide, *BaO* barium oxide, *CaO* calcium oxide, *Cl* chlorine, *Cr₂O₃*, chromium oxide, *CuO* copper oxide II, *Fe₂O₃* iron oxide III, *K₂O* potassium oxide, *MgO* magnesium oxide, *MnO* manganese oxide, *Na₂O* sodium oxide, *NiO* nickel oxide II, *P₂O₅* phosphorus pentoxide, *Rb₂O* rubidium oxide, *SiO₂* silica oxide, *SO₃* sulfur trioxide, *SrO* strontium oxide, *TiO₂* titanium oxide IV, *ZnO* zinc oxide, *C.V.* coefficient of variation

chemical characteristics between trees in the same site makes the overall variation between sites insignificant. The differences between trees may be attributed to genetic characteristics. Indeed, Zhang et al. (2015) showed that for

Populus tomentosa Carr. the wood chemical properties were under stronger genetic control than growth traits.

The proximate analysis of the Pyrenean-oak heartwood indicates an ash content of 0.23 ± 0.12 wt%, which is

Table 4 Analysis of variance of the proximate and ultimate analysis of the Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood samples (significance level of 0.05)

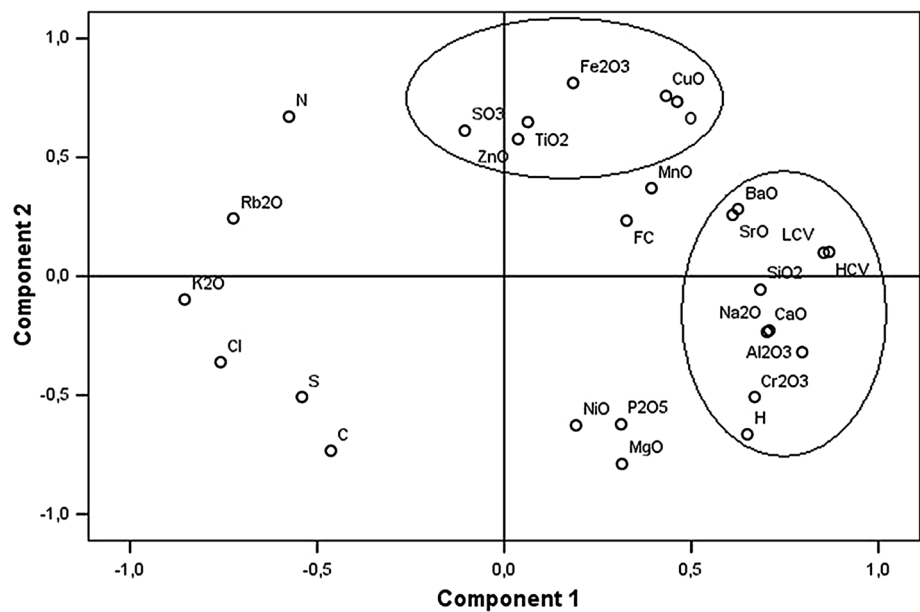
Source of variance	Ash	C	LCV	HCV	C	O	N	H	S	C/N	C/H
<i>Site</i>											
F	9.0	0.689	4.112	4.308	1.110	1.175	1.533	1.151	3.709	1.180	1.134
Sig.	0.052	0.617	0.138	0.131	0.467	0.449	0.367	0.455	0.155	0.447	0.460
<i>Tree</i>											
F	27.0	0.027	25.158	26.785	0.038	0.038	22.533	25.996	2.020	5.817	1.458
Sig.	0.014*	0.881	0.015*	0.014*	0.857	0.857	0.018*	0.015*	0.250	0.095	0.314

Table 5 Analysis of variance of the ash composition of the Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood samples (significance level of 0.05)

Source of variance	Al ₂ O ₃	BaO	CaO	Cl	Cr ₂ O ₃	CuO	Fe ₂ O ₃	K ₂ O	MgO
<i>Site</i>									
F	1.214	1.720	0.562	1.783	0.485	3.485	1.568	0.963	2.537
Sig.	0.439	0.333	0.676	0.323	0.716	0.166	0.360	0.512	0.232
<i>Tree</i>									
F	8.471	1.420	7.207	2.315	4.835	0.052	0.694	4.012	5.673
Sig.	0.062	0.319	0.075	0.226	0.115	0.834	0.466	0.139	0.097

Source of variance	MnO	Na ₂ O	NiO	P ₂ O ₅	Rb ₂ O	SiO ₂	SO ₃	SrO	TiO ₂	ZnO
<i>Site</i>										
F	7.278	1.095	0.356	1.157	0.381	1.703	0.989	1.068	5.402	1.970
Sig.	0.069	0.471	0.791	0.454	0.775	0.336	0.504	0.479	0.100	0.296
<i>Tree</i>										
F	1.008	4.249	0.533	1.256	3.117	3.524	1.111	0.778	2.545	1.308
Sig.	0.389	0.131	0.518	0.344	0.176	0.157	0.369	0.443	0.209	0.336

Fig. 1 Components 1 and 2 from the principal component analysis (PCA) for the chemical variables of the Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood



correlation between Ba wood concentration and soil bases. The wood elements' concentration can also be used to evaluate or monitor changes in soil and environment quality (Guyette and Cutter 1994; Poszwa et al. 2003; Augustin et al. 2005). For example, the Mn concentration in plants has been used as a biomarker for the switch of buffering mainly with the exchange of base cations and the dissolution of aluminum oxides (Hildebrand 1986; Ulrich 1991). Soil type and chemical characteristics should be taken into account too when designing a dendrogeochemical study of environmental change.

The average content of Mn found in the present study is $2583 \mu\text{g g}^{-1}$ DW, which is relatively high. Yang et al. (2015) reported Mn concentrations ranging from 300 to $800 \mu\text{g g}^{-1}$ for *Salix* clones, while in Mn-contaminated soils the concentrations varied between 1840 and $4572 \mu\text{g g}^{-1}$. *Pseudotsuga menziesii* showed maximum Mn concentrations of $3260 \mu\text{g g}^{-1}$ DW (stem) (Dučić et al. 2006), while in this study the maximum concentration was $5557 \mu\text{g g}^{-1}$. Augustin et al. (2005) reported concentrations ranging from 50 to $100 \mu\text{g g}^{-1}$ for Norway spruces, while in contaminated soils varied between 400 and $600 \mu\text{g g}^{-1}$. The Mn concentrations in the present Pyrenean-oak wood are also higher than those found in the wood from *Cassia siamea* (Cassia), *Azadirachta indica* (Neem) and *Holoptelia integrifolia* (Holoptelia) growing in contaminated soils (wood concentrations of Mn ranging from 626 to $1248 \mu\text{g g}^{-1}$) (Raju et al. 2008).

Conclusion

A study on the variation of the chemical composition of Pyrenean-oak (*Quercus pyrenaica* Willd.) heartwood among different sites and its relationship with the soil chemical characteristics is presented. The chemical characteristics of the wood were quantified in terms of proximate and ultimate analysis, calorific value and ash composition. Subsequently, the relationship between the wood chemical characteristics and the soil characteristics was also examined. The main conclusions from this study are as follows:

- We found no significant differences between the heartwood chemical characteristics from different sites.
- Significant differences between trees from the same site regarding the wood ash content, the calorific value and the contents of nitrogen and hydrogen were observed.
- Strong correlations were observed between the heartwood ash contents of BaO, MnO, CuO and TiO₂ and the soil nitrogen, phosphorous and potassium and the soil exchangeable base contents.

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