Valorization of residual biomass from forest management and wood processing for the production of bioactive compounds

A. Fernández-Agulló [1], M.S. Freire[1], C. Ramírez-López Ramallal[2], J. Fernández-Moya[2], J. González-Álvarez[1]

[1]Department of Chemical Engineering, Universidade de Santiago de Compostela, Santiago de Compostela, Galicia, 15782, Spain
[2]Department of Forestry, Bosques Naturales S.A., Avenida de la Vega 1, edificio 3, planta baja, Alcobendas, Madrid, 28108, Spain
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Presenting author email: julia.gonzalez@usc.es

Wood is a sustainable material which is available in large quantities and easy to produce. However, it should be guaranteed that trees grow in well-managed forests and that the techniques used for its processing also assure a sustainable production. Based on the principles of circular economy, renewable resources must be used in a sustainable and circular way and residues minimized or completely removed by recycling or re-using (Herrero and Ibañez, 2018). Forest management and wood processing for obtaining different products such as timber for wood construction, cellulose pulp, etc., produce various residues that should be viewed as business opportunities as can represent valuable raw materials for obtaining different products or as energy sources. One of the alternatives that is leading to a considerable investigation is the search for natural materials that can be sources of safe antioxidant compounds for various industrial applications without leading to overexploitation of natural resources.

Bosques Naturales S.A. is a Spanish company that has plantations of trees of high economic value such as walnut (*Juglans major 209 x Juglans regia*) with selected plant materials and reproduced *in vitro*. In Juglans species gallic acid and various ellagitannins have been found in wood extractives, naphtoquinones and flavonoids in bark extractives (Burtin *et al*,1998) and various phenolic acids and flavonoids in leaf extractives (Amaral *et al*, 2004). In the stages of forest maintenance and initial wood processing, various biomass residues are produced. In this work, we planned to analyze the capacity of some of these residual materials obtained from the above mentioned species, namely, pruning wastes (leaves and branches), wood shavings and edged cuts (wood and bark) as sources of antioxidant compounds that could be valuable in the cosmetic, pharmaceutical, nutraceutical or food industries. With the aim of developing green extraction processes (Chemat *et al*, 2012) water and aqueous solutions of ethanol, a generally recognized as safe (GRAS) solvent, were selected as solvents, together with the use of raw materials obtained from controlled crops.

The operational conditions involved in the extraction process were analyzed in order to find those conditions that lead to high extraction yields together with high antioxidant activities of the extracts. A Plackett-Burmann experimental design was applied to analyse the influence of temperature (40 and 80ºC), liquid/solid ratio (5/1 and 10/1 mL/g for edged cuts and pruning wastes, and 10/1 and 15/1 mL/g for shavings), time (30 and 90 min) and aqueous ethanol concentration (0 and 50%) on extraction yield and extract properties. The extractions (approximately 5 g of material) were carried out in duplicate in an orbital shaking bath with temperature control at a shaking speed of 90 rpm. Under the operational conditions selected for each material, experiments were carried out at a larger scale (approximately 500 g of material) in a temperature-controlled reactor with mechanical stirring. The extracts were analysed to determine the total phenols content (TPC) (Folin-Ciocalteau method) and the antioxidant activity (FRAP, DPPH and ABTS methods). In addition, a selection of the extracts was analysed by UPLC/ESI-Q-TOF-MS to identify and quantify the compounds with potential antioxidant activity present.

Table 1 shows a summary of the results of the experimental design for all the fractions analysed. Comparing walnut fractions, the greater antioxidant capacities and TPC together with relatively high extraction yields were obtained for shavings.

Table 1. Summary of the results of the experimental design for all walnut fractions analyzed.

	Extraction yield (%)	***FRAP antioxidant activity (nmol AAE/mg extract)***	***Total phenols content (g GAE/100 g extract)***
Shavings	3.43-7.04	1907-2915	21.70-38.74
Edged cuts	4.24-9.75	1006-1760	14.53-30.86
Pruning wastes	3.03-6.26	533-1703	8.80-26.28

Temperature and ethanol concentration were the most significant variables for all the fractions studied and, in general, increasing both variables extraction yield and extract properties improved. 50% EtOH, 80ºC and the lowest L/S essayed, the most economical one, were selected for performing the large-scale extraction experiments. Fig. 1 shows the results obtained for the DPPH radical inhibition percentage of the extracts comparatively with those obtained with water as solvent using Trolox as reference antioxidant. As observed, the 50% EtOH shavings extract presents an inhibition behavior very close to that of the commercial antioxidant.

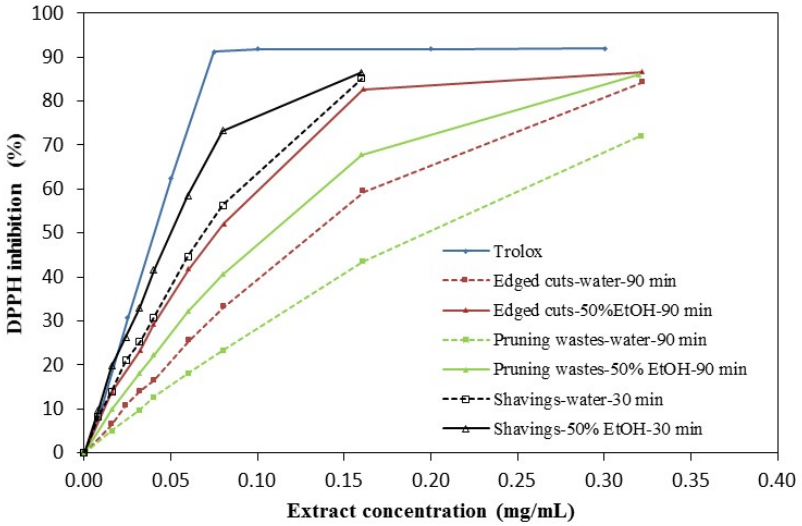

Fig. 1. Percentage of DPPH radical inhibition against the concentration of extracts of various walnut fractions.

All walnut extracts showed the same phenolic profile. Gallic, ellagic and ferulic acids were the major phenolic acids, and flavonol quercetin-3-β-D glucoside the main flavonoid. The 50% EtOH extract of the edged cuts showed the highest content in quercetin-3-β-D glucoside that was the main compound both in this extract and in the aqueous one. The major contents in gallic and ellagic acids corresponded to the aqueous and 50% EtOH extracts of shavings, respectively, being also their main compounds. Ferulic acid was found in similar amounts in the aqueous extracts of the three materials, being slightly higher in shavings.

In conclusion, this study contributed to the valorization of various walnut biomass residues as sources of valuable compounds such as phenolic acids and flavonoids with bioactive functions and potential applications in the food, chemical, and pharmaceutical industries. In addition, the proposal constitutes an environment friendly alternative for the disposal of these biomass residues.

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