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VALORIZATION OF MOROCCAN OLIVE STONES BY USING IT IN PARTICLEBOARD PANELS

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ABSTRACT

The main objective of this work was to find new applications to valorize olive stones (endocarp and seed). In order to improve knowledge on olive stones, the phenolic compounds concentration of three varieties of Moroccan olive trees: Moroccan Picholine, Menara and Haouzian were studied. Olive stones of three varieties were characterized by Fourier Transform Mid Infrared Spectroscopy (FT-MIR). Total phenolic compounds are quantified after solid-liquid extraction by an assay of Folin-Ciocalteu. Moroccan Picholine stones (11.32 mg GAE/g DM) have a higher content of total phenolic compounds than Haouzia stones (4.55 mg GAE/g DM) and Menara stones (3.56 mg GAE/g DM). Thermogravimetric analysis indicates that up to 195°C; there is no degradation of the stones. The biocide performance on agar-agar was tested with decay fungi. Biodegradation studies show that the most interesting results are obtained with Moroccan Picholine stones. The presence of Moroccan Picholine in a particleboard panels improves the total resistance of the particleboard panels against both *Coriolus versicolor* and *Coniophora puteana* rot fungi.

Keywords: Decay resistance; olive stone; particleboard; phenolic compounds.

INTRODUCTION

Oleaceae is a family comprising 600 species in 25 genera. Many of the genera are economically important such as the olive (*Olea europaea*) which is cultivated for its fruit and oil (Wallander and Albert 2000). The olive tree is among the oldest woody crops and is particularly widespread throughout the Mediterranean region and plays an important role in its rural economy, local heritage, and environment protection. The largest producing countries are located in the Mediterranean and Middle East regions providing 98% of the total cultivated surface area, and 99% of the total olive fruit production (Niaounakis and Halvadakis 2006, Besnard *et al*. 2011).

Olea europaea dried fruit has pharmacological properties, such as anti-bacterial, anti-viral, anti-inflammatory activities and detoxification (Ding 1999). Such properties have been attributed to the presence of many compounds including phenols. Olive fruits are rich in phenolic compounds that represent 1 to 14% weight of dry pulp. The phenolic composition in the various parts of the fruit is complex depending on the variety, collection season, growing conditions, and time of ripening (Solinas *et al.* 1975, Vlahov 1992, Esti *et al.* 1998, Romani *et al*. 1999). Very few studies have

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focused upon the phenolic composition of olive seeds, nevertheless some phenolic compounds have been identified including salidroside, nuzhenide, hydroxytyrosol, nuzhenide 11-methyl oleoside, oleuropein, tyrosol, and demethyloleuropein (Fig. 1) at all stages of maturation (Maestro-Durán *et al.* 1994, Servili *et al.*1999, Silva *et al.* 2010).

The olive stones and seeds are an important by-product generated in the olive oil extraction. Generally, this material is used as combustible to produce electric energy or heat. A large number of research articles have been published dealing with the chemical composition of olives and olive oil. However, only a few studies have been dedicated to analyzing the components and uses of the olive stone (Rodríguez *et al.* 2008).

The main objectives of this work are the extraction of phenolic compounds, the characterization (chemical, thermal and biological) and the valorization of Moroccan olive stones in particleboard panels.

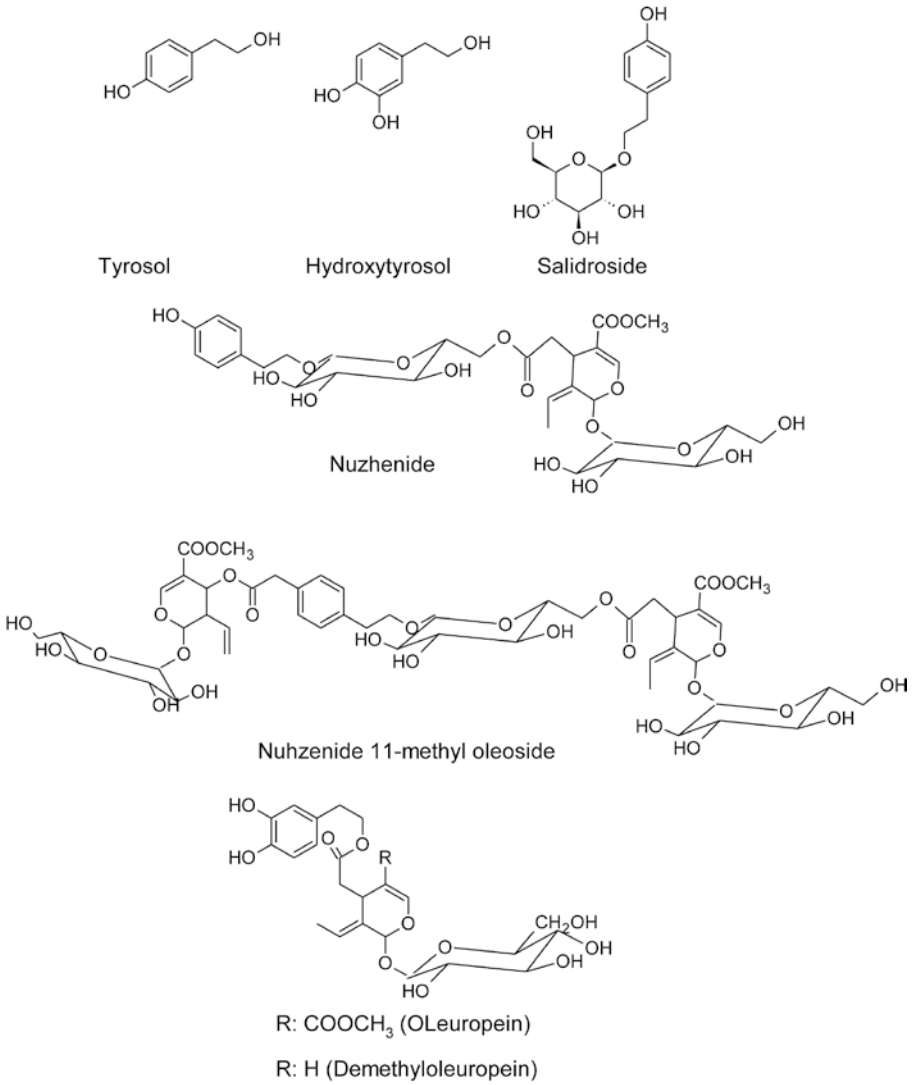

Figure 1. Some components found in stone and seed olives.

EXPERIMENTAL METHODS

Plant materials

Mature olive fruits were obtained from the Moroccan Picholine variety, which grows widely in the province of Beni Mellal and from Menara and Haouzia varieties growing in the province of Marrakech of Middle Atlas and Marrakech-Tensift-Al Haouz (Morocco), respectively. Olive fruits were boiled (between 70 and 80°C) in water for 8 minutes, and pulped by crushing them manually. Olive stones were then air dried on paper towels (2-4 days). After drying, the olive stones were ground (particle less than 0.5 mm) in a knife mill "Retsch SK1".

Maritime pine (*Pinus pinaster*) particles was provided by the sawmill Ets. Labadie (Roquefort, France) Sd (moisture content around 8–10%).

Extraction and estimation of total phenolic compounds

The solid-liquid extraction of total phenolic compounds was performed with a Soxhlet apparatus. Olive stone powder (60 g) was extracted in a Soxhlet apparatus, first with hexane (500 ml) for 5 hours to remove lipids, then with acetone (500 ml) for 5 hours and finally with ethanol (500 ml) for 5 hours to remove total phenolic compounds (Rakib *et al.*2010).

Total phenols are estimated by Folin-Ciocalteu method (Singleton and Rossi 1965, Scalbert *et al.* 1989). A 2.5 ml portion of Folin-Ciocalteu reagent and 2 ml of a sodium carbonate solution (75 g/l) are added to 0.5 ml of the diluted extract. The assay tubes are kept 15 min in a water bath at 50°C and then transferred to cold water. The absorbance read was 760 nm. A calibration curve with equation: $y = 0.0021x + 0.0015$ ($r^2 = 0.99$) was constructed using gallic acid solutions within the range 10–100 mg/l. Contents of total phenolic compound in olive stones were expressed as gallic acid equivalents in milligrams per gram dry matter (mg GAE/g, DM). The results were averages of triplicate analyses.

Fourier Transform Middle-Infrared Spectroscopy (FT-MIR)

Analyses were performed with FT-MIR. The spectra of the samples were recorded with a Perkin Elmer's FTIR (Spectrum One). Samples were deposited without preparation on attenuated total reflection (ATR) cell that is equipped by a diamond crystal. The crystal was cleaned between measurements by deionized water and dried by lint-free tissue. Spectra were recorded between 4000 and 600 cm^{-1}, at 0.5 cm^{-1} nominal resolution.

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was used to determine the thermal stability and degradation of olive stones using a TGA Q50 thermogravimetric apparatus. Ten milligrams of each cured sample were placed on a balance located in the furnace and heat was applied over the temperature range from room temperature to 600 °C at a heating rate of 5 °C/min in air. Mass losses versus temperature thermograms were obtained showing the different decomposition processes.

Particleboard preparation

Single layer laboratory particleboards of dimension 350 mm x 310 mm x 14 mm were prepared from maritime pine (*Pinus pinaster*). The addition of the commercial Urea Formaldehyde (UF) resins to the particles was 7.5 % based on solid mass; press time was 7.5 min at 195°C press platen temperature with a maximum specific pressure of 25 kg/cm^2. The UF resin had a density of 1280–1290 kg/m^3, a pH of about 8.5–9, and a viscosity of 350–600 mPa.s, all measured at 20 ± 2°C; the

gel time was 40-50 seconds at 100°C. The content of free formaldehyde is maximum 0.15 % based on the liquid resin. The particles with size between 1 to 4 mm were dried to approx 2 ± 0.5 % moisture content prior to application of the resin. Each olive stone powder was ground and mixed at different concentrations (from 0 to 15%, w/w) in particles. The target board density was 710 kg/m^3. The particleboards were pre-conditioned at 25°C and 65 % relative humidity in a Vötsch climate room for one week before testing.

Biological properties

Decay test of the particleboard composites

Particleboards composites measuring 20 × 20 × 14 mm^3 were prepared for decay test, from Maritime pine (*Pinus pinaster*). A fungal decay test was done according to an adaptation of the standard EN 113 (AFNOR 1996) using a brown rot fungus, *Coniophora puteana* (BAM Ebw.15) and the white rot fungus, *Coriolus versicolor* (CTB 863A). A culture medium was prepared in Roux flasks. In each one, a nutritive medium, made up of a malt-agar mixture was placed (40 g of malt and 20 g of agar dissolved in 1000 ml distilled water). After sterilization (121°C for 20 min at 1 bar), the *Coriolus versicolor* and *Coniophora puteana* were inoculated on culture medium in Petri dishes under sterile conditions. Then they were placed in constant conditions at 25°C and 65% relative humidity for 3 weeks to favour fungal development. After being conditioned at 25°C and 65% air moisture content, the particleboard composite samples were put into Petri dishes and exposed to *Coriolus versicolor* and *Coniophora puteana* for 16 weeks in darkness. Finally, test results were expressed as percentage of weight loss of composite panels due to fungal attacks after decay test using Equation (1) the measurement was done after drying of each sample). Ten replicates were used for each decay fungus. Weight loss was calculated as follows:

$$\text{Weight loss (\%)} = \left[\frac{m_i - m_f}{m_i}\right] 100 \qquad (1)$$

Where m_i and m_f are the oven-dry weights of the sample before and after the decay test.

Decay test of olive stone powders

In order to confirm the fungicidal effect, olive stone powder was added to the culture medium. Each olive stone powder was ground and mixed at different concentrations (from 0 to 7%, p/v) in malt-agar culture medium. After sterilization, the *Coriolus versicolor* and *Coniophora puteana* fungi were inoculated in the centre of the culture medium under sterile conditions. Then they were placed in constant conditions at 25°C and 65% relative humidity for 15 days to favour fungal development. The mean radius of the fungal development was measured. Test results were expressed as percentage of fungal development using Equation (2). Ten replicates were used for each fungal development.

$$\text{Percentage of fungal development (\%)} = \frac{R_1}{R_0} 100 \qquad (2)$$

Where

R_1 : is the mean radius of the fungal development after 15 days (olive stone powders present in Petri dishes).

R_0 : is the mean radius of the fungal development after 15 days (control: without presence of olive stone powder).

RESULTS AND DISCUSSION

Impact of olive stones varieties on polyphenols extraction

Table 1 shows the polyphenols concentration in acetone and ethanol extracts. The results showed that Moroccan Picholine variety growing in the province of Beni Mellal had the higher contents of total phenolic compounds (11.32 mg/g DM), followed by Haouzia (4.55 mg/g DM) and Menara (3.56 mg/g DM) variety. The relative amounts of phenols found in our experiment are in agreement with those already reported by some authors which described the lower levels of phenols in stones and seeds in comparison to the remaining tissues (pulp, leaves) of olives (Ryan *et al*. 2003). Nevertheless, compared with some other berries, the total phenolics contents in olive stones (3.56-11.32 mg/g DM) are higher than those of Oregon canberries (4.95–9.80 mg/g DM) berries (Wada and Ou 2002) and bayberries (3.60-4.46 mg/g DM) (Zhongxiang *et al*. 2007).

Table 1. Polyphenol contents of various olive stones. Three replicates of each sample. S.D: standard deviation.

Olive varieties	Extraction methods		Total phenolics (mg GAE/g DM)
	Ethanol extract (mg GAE/g DM) Mean ± S.D	Acetone extract (mg GAE/g DM) Mean ± S.D	
Moroccan Picholine	3.07 ± 0.007	8.25 ± 0.023	11.32
Menara	1.86 ± 0.001	1.70 ± 0.002	3.56
Haouzia	2.15 ± 0.003	2.40 ± 0.015	4.55

FT-MIR spectroscopy can be used to address qualitative and quantitative analyses. In this view, comparison with literature data provides preliminary information for the compositional characterization of FT-MIR bands of olives stones samples. Spectra of different olives stones are presented in figure 2. For the three varieties the spectra seem very similar. By means of FT-MIR spectroscopy, the original structure of different polyphenols can be clearly distinguished. The peak around 1285 cm^{-1} indicates a characteristic feature for the flavonoid-based tannins. This peak was assigned to the ethereal C-O stretching vibration (Socrates, 2001, Edelmann and Lendl 2002) arising from the pyran-derived ring structure of this class of tannins. Two broader peaks at around 1350 cm^{-1} and between 1290 and 1150 cm^{-1} can be observed in the spectra of gallic acid, tannic acid (gallotannin) and in the ellagitannin preparation with shifts in exact peak position and deviations in peak shape. Both peaks can be assigned to the combination of C-O stretching and O-H deformation vibrations (Socrates 2001, Edelmann and Lendl 2002).

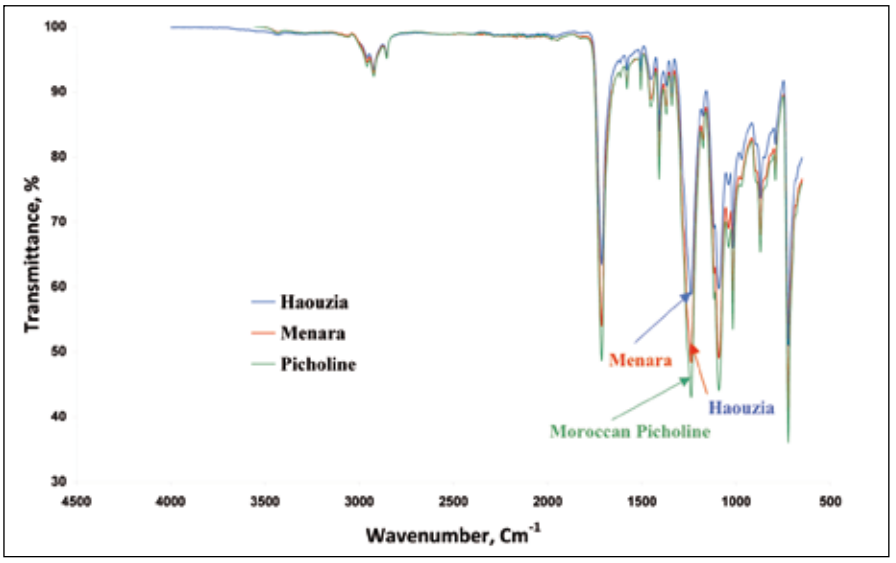

Figure 2. IR spectra of Haouzia, Menara and Moroccan Picholine olive stones.

Thermal study of different olive stones

Thermogravimetric analysis can check the thermal decomposition and thermal stability of the olive stones. Figure 3 shows the TGA curve of the Moroccan Picholine, Menara and Haouzia varieties in air atmosphere at a heating rate of 5°C/min, result that matches to typical findings for the decomposition of other lignocellulosic materials. Two steps can be differentiated in the TGA curves: I- Up to 200 °C, the weight loss corresponds to moisture release; II- The main weight loss takes place between 200 and 400°C. In this temperature range, two decomposition processes are well defined, with maximum weight loss rates appearing at 245 and 330°C. The first step can be attributed to the group of reactions involved in hemicellulose degradation (Órfão *et al.* 1999, Williams and Besler 1993), and the second one to those related to the thermal decomposition of cellulose (Órfão *et al.* 1999, Williams and Besler 1993).

The curve also shows that thermal degradation began to occur only after the materials have absorbed certain amounts of heat energy. Heat initiated the degradation processes and the breakdown of the structure by causing molecular chain ruptures. The results obtained with thermogravimetric analysis confirm that at 195 °C they risk no degradation of the olive stones.

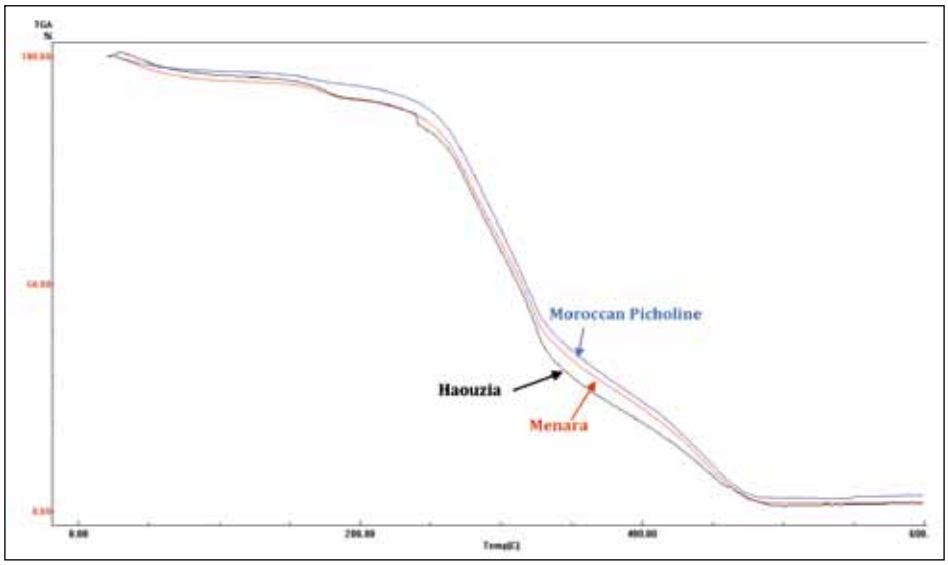

Figure 3. TGA graphical analysis for Menara, Haouzia and Moroccan Picholine olive stone varieties

Decay resistance of the particleboard composites

Weight losses of the particleboard composites caused by fungal decay after 16 weeks are shown in table 2. We can see that the control particleboard was attacked by both decay fungi (*Coriolus versicolor* and *Coniophora puteana*) with weight losses near 40%. This proves that the fungus was virulent and the test of durability was valid according to the relevant European Norm 113 (EN 113, 1996). The values of weight losses for all other particleboard panels decreased. Laboratory tests have shown that the addition of olive stones (5, 10 and 15%) is very effective in preventing attack by both decay fungi. We also observed that particleboard with Moroccan Picholine (5, 10 or 15%) showed considerable resistance to both the decay fungi compared with particleboard with Haouzia (5, 10 or 15%) and Menara (5, 10 or 15%).

Results in table 2 seem to indicate that *Coniophora puteana* could be more efficient in wood degradation than *Coriolus versicolor*. This may be due to the differences between their decay mechanisms. This tendency has been previously observed by Nemli *et al.* (2006) and Moubarik *et al.* (2009).

Table 2. Weight loss of composite in presence of olive stones after exposure to both decay fungi (*Coriolus versicolor* and *Coniophora puteana*). Ten replicates of each decay fungus. S.D: standard deviation.

Olive varieties	Samples	Weight loss (%)	
		Coriolus versicolor Mean ± S.D	*Coniophora puteana* Mean ± S.D
Moroccan Picholine	particleboard + 0% (Control)	35 ± 3.1	41 ± 2.8
	particleboard + 5%	24 ± 5.2	28 ± 3.6
	particleboard + 10%	19 ± 4.1	23 ± 2.5
	particleboard + 15%	12 ± 2.2	18 ± 3.7
Menara	particleboard + 0% (Control)	35 ± 3.1	41 ± 2.8
	particleboard + 5%	32 ± 4.4	37 ± 3.2
	particleboard + 10%	27 ± 5.1	33 ± 4.7
	particleboard + 15%	22 ± 3.7	28 ± 3.7
Haouzia	particleboard + 0% (Control)	35 ± 3.1	41 ± 2.8
	particleboard + 5%	30 ± 5.1	35 ± 5.2
	particleboard + 10%	25 ± 6.2	30 ± 4.9
	particleboard + 15%	19 ± 4.7	25 ± 3.6

Fungicide effect of olive stones

Table 3 shows the effect of olive stones (Picholine, Haouzia and Menara) concentration on fungal development. A decrease in fungal development for both decay fungi *Coriolus versicolor* and *Coniophora puteana* is observed on incorporation of olive stones into the culture medium. The results in table 3 confirm the fungicide effect of olive stones that contributes to improve the total resistance of the particleboard composites.

Hart and Hillis (1972) have demonstrated that the presence of phenolic compounds improves the durability of wood. Further studies evaluating the fungicide effect of phenolic compounds (tannins) also confirmed this activity (Pizzi and Conradie 1986, Charrier *et al.* 1995, Aloui *et al.* 2004, Cornelius *et al.* 2004, Moubarik *et al*. 2009).

Table 3. Effect of olive stone concentrations on fungus development. Ten replicates of each fungal development. S.D: standard deviation.

Olive varieties	Concentration (%)	Percentage of fungus development (%)	
		Coriolus versicolor Mean ± S.D	*Coniophora puteana* Mean ± S.D
Moroccan Picholine	0	100 ± 0	100 ± 0
	2	20 ± 0.5	25 ± 0.7
	5	6 ± 0.1	8 ± 1
	7	1 ± 0	3 ± 0.3
Menara	0	100 ± 0	100 ± 0
	2	29 ± 1.1	31 ± 1.4
	5	14 ± 0.9	17 ± 1.1
	7	7 ± 0.2	9 ± 0.4
Haouzia	0	100 ± 0	100 ± 0
	2	26 ± 0.9	28 ± 1.7
	5	12 ± 1.1	15 ± 1.2
	7	5 ± 0.6	7 ± 0.4

CONCLUSION

The present work proposes an innovative valorization of olive stones for particleboard production. The result of polyphenols extraction from Moroccan Picholine variety growing in the province of Beni Mellal had the higher contents of total phenolic compounds (11.32 mg/g DM), followed by Haouzia (4.55 mg/g DM) and Menara (3.56 mg/g DM) variety. The thermogravimetric analysis shows no significant degradation of the three olive stones varieties at temperature less than 195 °C. A good correlation was observed between the resistance to fungi and the concentration of olive stones. A decrease in fungal development for both decay fungi *Coriolus versicolor* and *Coniophora puteana* is observed after olive stones incorporation into the culture medium. The results of the present work contribute to the valorization of olive stones in the Mediterranean countries and especially in Morocco that has an ambitious agricultural strategy called «Green Morocco».

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