

Waste valorization in winemaking industry: Vine shoots as precursors to optimize sensory features in white wine

Maria Calderon-Martin^a, Esperanza Valdes-Sanchez^b, Maria F. Alexandre-Franco^a, M. Carmen Fernandez-Gonzalez^a, Mar Vilanova de la Torre^c, Eduardo M. Cuerda-Correa^{a,*}, Vicente Gomez-Serrano^a

^aDepartamento de Química Orgánica e Inorgánica, Facultad de Ciencias, Universidad de Extremadura, Avenida de Elvas s/n, 06006-Badajoz, España

^bArea de Enología. Instituto Tecnológico Agroalimentario de Extremadura (CICYTEX-INTAEX) Government of Extremadura. Avda Adolfo Suarez s/n, 06007 Badajoz, España

^cInstituto de Ciencias de la Vid y del Vino (ICVV) Consejo Superior de Investigaciones Científicas CSIC-Universidad de La Rioja-Gobierno de La Rioja, Carretera de Burgos km 6, 26007 Logroño, España

SUPPLEMENTARY MATERIAL

Table S1.- Operational conditions of carbonization and physical and chemical activation processes.

Carbonization							
Substrate	Carbonization temperature/°C	Atmosphere	Flow/ mL/min	Activation temperature/°C	Activation time/h	Yield/ %	Notation
VS	600	N ₂	80	-	2h	28.3	C600
VS	900	N ₂	80	-	2h	25.1	C900
Physical activation							
Substrate	Carbonization temperature/°C	Atmosphere	Flow/ mL/min	Activation temperature/°C	Activation time/h	Yield/ %	Notation
C600	-	Air	10	300	1	79.4	A
C900	-	CO ₂	10	750	1	82.6	CD
C900	-	N ₂ -steam	80(N ₂)	700	1	75.9	S
Chemical activation							
Substrate	Impregnation temperature/°C	Activating agent	Impregnation ratio	Carbonization temperature/°C	Carbonization time/h	Yield/ %	Notation
VS	85	H ₃ PO ₄	1:1	500	2	32.1	PA
VS	85	ZnCl ₂	1:1	500	2	39.5	ZC
VS	85	KOH	1:1	800	2	12.3	PH

Some remarks on the yield of the carbonization and activation processes

As summarized in Table S1, the yields of the pyrolysis process of VS is equal to 28.28% at 600°C and 25.08 % at 900°C. The fact that the carbonization temperature does not exert any significant influence on the final yield of the process is coherent with the mechanism commonly postulated for the pyrolysis of lignocellulosic materials. Briefly, when these materials are subjected to heat treatments under an inert atmosphere, a remarkable mass loss occurs at temperatures below 660°C due to the removal of volatile matter. Above this temperature, a progressive aromatization of the remaining carbonaceous solid can be observed. Such an aromatization takes place as a result of the release of a small amount of hydrogen. Hence, the mass loss is scarce and virtually constant regardless of what the thermal treatment temperature is.

The yield values corresponding to the physical activation process are also shown in Table S1. Mass losses take place because of the partial gasification of carbon atoms by reaction with the activating agent. Hence, the mass loss (and, consequently, the yield of the physical activation) depends on the activating agent used (namely, air, CO₂, or steam). The sequence of variation of the yield is $S < A < CD$. This fact influences not only the porous texture of the AC but also the adsorption process of solutes in aqueous solution. In general, for a given activating agent, an increase in the burn-off percentage usually results in the creation of broader pores since the widening of narrower pores is promoted.

On the other hand, the yield data corresponding to the preparation of AC by chemical activation are also presented in Table S1. Yield varies according to the series $ZC > PA \gg PH$. The relatively low yield values can be attributed to the release of volatile matter when the impregnated products are subjected to thermal treatment during the preparation of the ACs. The resulting lower yield for the KOH is in line with the carbonization temperature, which is much higher (800°C, instead of 500°C) in the case of the product impregnated with this activating agent. The low yield for sample PH is also

related to an intense chemical activity of potassium compounds at high temperatures.

The elemental analysis data of vine shoots are summarized in Table 2. These results indicate that this lignocellulosic material has relatively high contents of carbon, oxygen and hydrogen, and low contents of nitrogen and sulfur. The low sulfur content (0.05 %) makes VS a very attractive material to be used as a precursor for the preparation of ACs, since the release of sulfur exerts a negative effect on the environment. The low content of inorganic matter is also very interesting since inorganic matter may interfere with certain processes, such as adsorption of solutes.

Table S2. Correlation coefficients (r^2) found from regression analysis (95% significance level) between textural characteristics of the ACs and the values of polyphenolics and chromatics characteristics obtained in treated wines cv. Pardina at doses of 0.50 g L⁻¹

Variables	PI	H	F	BP	L*	C*	SBET	Wo	Vma-p	Vme-p	ρ Hg	VT
PI	1	0.948	0.998	0.713	0.014	0.590	-0.290	-0.280	-0.379	-0.227	-0.239	-0.557
H	0.948	1	0.945	0.758	-0.065	0.647	-0.393	-0.392	-0.423	-0.360	-0.185	-0.679
F	0.998	0.945	1	0.687	0.037	0.608	-0.297	-0.287	-0.379	-0.221	-0.213	-0.555
BP	0.713	0.758	0.687	1	0.122	0.226	-0.452	-0.449	-0.093	-0.081	-0.423	-0.338
L*	0.014	-0.065	0.037	0.122	1	-0.352	0.196	0.206	-0.106	0.633	0.502	0.204
C*	0.590	0.647	0.608	0.226	-0.352	1	-0.540	-0.526	-0.113	-0.545	-0.285	-0.489
SBET	-0.290	-0.393	-0.297	-0.452	0.196	-0.540	1	0.999	-0.554	0.232	0.630	-0.043
Wo	-0.280	-0.392	-0.287	-0.449	0.206	-0.526	0.999	1	-0.546	0.242	0.619	-0.033
Vma-p	-0.379	-0.423	-0.379	-0.093	-0.106	-0.113	-0.554	-0.546	1	0.370	-0.558	0.824
Vme-p	-0.227	-0.360	-0.221	-0.081	0.633	-0.545	0.232	0.242	0.370	1	0.293	0.765
ρ Hg	-0.239	-0.185	-0.213	-0.423	0.502	-0.285	0.630	0.619	-0.558	0.293	1	-0.127
VT	-0.557	-0.679	-0.555	-0.338	0.204	-0.489	-0.043	-0.033	0.824	0.765	-0.127	1

PI. H. F. BP. L*. and C*: Polyphenol Index. Hidroxycinamates. Flavonoids. Brown polymers. Lightness. Yellowness and Chromaticity respectively

Vme-p. VT. Vma-p. Wo. SBET. and ρ Hg: mesopore volume, total pore volume, macropore volume, micropore volume, specific surface area, and mercury density of activated carbons

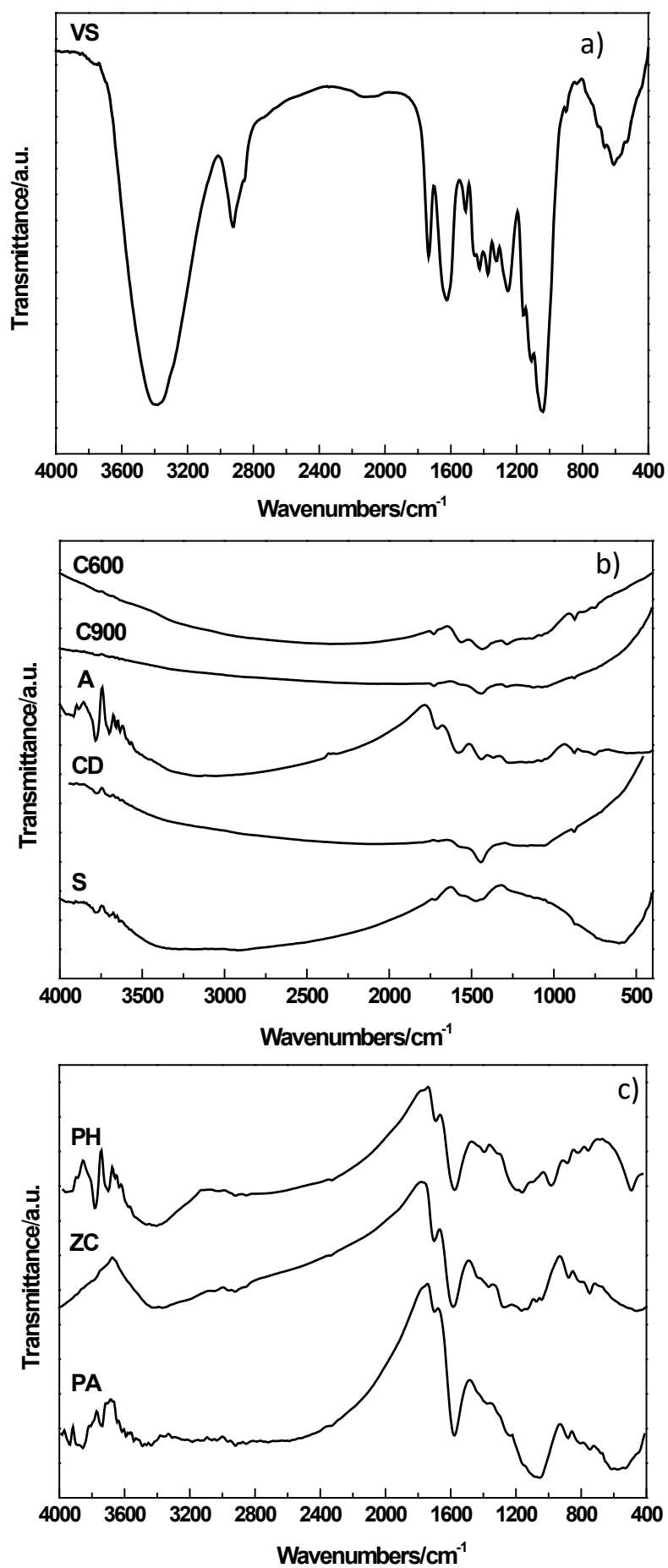


Figure S1: FT-IR spectra of vine shoots (a) and samples prepared by physical (b) and chemical (c) activation processes.