

Study of odorant compounds and sensory changes associated with wine spirit ageing using chestnut wood and Limousin oak under different technologies

Ilda Caldeira,^{1,2} Ofélia Anjos, ^{3,4} Cláudia Vitória,³ Tiago A. Fernandes,^{5,6} Sofia Catarino,^{7,8} Sara Canas^{1,2}

¹Instituto Nacional de Investigação Agrária e Veterinária, Polo de Dois Portos, Quinta de Almoinha, 2565-191 Dois Portos, Portugal; ilda.caldeira@iniav.pt; sara.canas@iniav.pt;

²*MED* – *Mediterranean Institute for Agriculture, Environment and Development & CHANGE* – *Global Change and Sustainability Institute, Instituto de Investigação e Formação Avançada, Universidade de Évora, Pólo da Mitra, Ap. 94, 7006-554 Évora, Portugal.* ³*Instituto Politécnico de Castelo Branco, Quinta da Senhora de Mércules, 6001-909 Castelo Branco, Portugal; ofelia@ipcb.pt; adriana-1998@hotmail.com*

⁴Centro de Estudos Florestais, Instituto Superior de Agronomía, Universidade de Lisboa, Tapada da Ajuda, 1349-017 Lisboa, Portugal. ⁵Centro de Química Estrutural, Associação do Instituto Superior Técnico para a Investigação e Desenvolvimento (IST-ID), Universidade de Lisboa, 1049-001 Lisboa, Portugal; tiago.a.fernandes@tecnico.ulisboa.pt

⁶Departamento de Ciências e Tecnologia, Universidade Aberta, Rua da Escola Politécnica,141-147, 1269-001 Lisboa, Portugal. ⁷LEAF—Linking Landscape, Environment, Agriculture and Food Research Center, Associated Laboratory TERRA, Instituto Superior de Agronomia, Universidade de Lisboa, Tapada da Ajuda, 1349-017 Lisboa, Portugal;

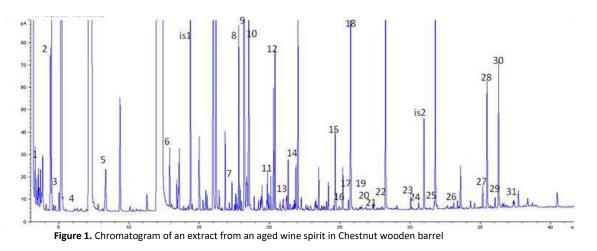
⁸ CEFEMA – Center of Physics and Engineering of Advanced Materials, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1, 1049-001 Lisboa, Portugal

Wine spirit, resulting from wine distillation, usually undergoes ageing in wood before being marketed. Traditionally, the oak wood, especially from the French region of Limousin (mostly *Quercus robur L.*), is used for manufacturing the barrels for this purpose. However, the results from several studies pointed out the suitability of chestnut barrels for the ageing of wine spirits¹. Usually, this ageing process is carried out by placing the distilled beverage in wooden barrels for a more or less extended time. However, recent works² showed that alternative systems, in which wood fragments are placed into the beverage kept in stainless steel tanks, are an interesting technology. Some of these works also highlighted the importance of oxygen, providing promising results through the combination of staves with micro-oxygenation in the ageing of these beverages^{2,3}.

This work aimed to study different levels of micro-oxygenation applied simultaneously with wood staves in the ageing of wine spirit in glass demijohns and their comparison with the traditional system using wooden barrels. A two way experimental design was established with two levels of the first factor (Chestnut wood *versus* oak wood from Limousin) and five levels of the second factor (different micro-oxygenation conditions) and two replicates of each assay modality, giving a total of 20 experimental units. The same wine spirit produced in the Lourinhã region (Portugal) was used to fill these units. The ageing process was followed for 12 months, and samples were collected over time. The volatile composition of the samples was evaluated by GC-FID and GC-MS. After 12 months, the wine spirits were bottled and analyzed regarding their volatile composition (GC-FID and GC-MS) and sensory profile, which was carried out by a trained sensory panel. Statistical analysis (ANOVA and principal components analysis-PCA) were applied to the chemical and sensory results.

The volatile determination was focused on the compounds (Fig. 1) previously assigned as odorant compounds.





1: Ethyl isobutyrate; 2: isobutyl acetate; 3: ethyl butanoate; 4: ethyl isovalerate; 5: isoamyl acetate; 6: ethyl hexanoate; pi1: 5-methyl-2-hexanol (internal standard 1); 7: trans-2-hexenol; 8: ethyl octanoate 9: acetic acid; 10: furfural; 11: linalool; 12: 5-methylfurfural; 13: butanoic acid; 14: isovaleric acid; 15: hexanoic acid; 16: guaiacol; 17: trans β-methyl-γ-octalactone; 18: 2-phenylethanol; 19: *cis* β-metil-γ-octalactone; 20: 4-methylguaiacol; 21: 4-ethylguaiacol; 22: diethyl malate; 23: eugenol; 24: 4-ethylphenol; pi2: 3,4- dimethylphenol (internal standard 2); 25: syringol; 26: 4- methylsyringol; 27: dodecanoic acid; 28: HMF; 29: 4- allylsyringol; 30: vanillin; 31: acetovanillone.

The obtained results confirmed the significant influence of the wood's botanical species on the volatile composition and the sensory profile of the aged wine spirits. Those aged with chestnut wood had significantly higher overall quality than those obtained with Limousin oak wood. Regarding the ageing system, the wine spirits aged by alternative system seemed to present higher contents of some odorant compounds, namely some volatile phenols. Particularly, the modality of highest flow of micro-oxygenation (2 mL/L/months during 60 days followed by 0.6 mL/L/months during the remaining ageing time) resulted in the best sensory results.

Acknowledgements: The authors are very grateful to the sensory panel. The authors also thank Victor de Freitas as the Scientific Consultant of the Project POCI-01-0145-FEDER-027819, and to João Pedro Catela, Nádia Santos, Manuela Gomes, Eugénia Gomes and Inês Antunes from Adega Cooperativa da Lourinhã, José Abílio Gonçalves and Sérgio Gonçalves from Tanoaria J. M. Gonçalves, Ana Partidário from INIAV—Oeiras, Pedro Rodrigues and Diogo Rodrigues from AZ3Oeno Portugal, Sílvia Lourenço, João Amaral, Deolinda Mota from INIAV—Polo de Dois Portos, and A. Pedro Belchior for their technical support.

Funding: This work is funded by Portuguese National Funds throughout FCT—Foundation for Science and Technology under the Project POCI-01-0145-FEDER-027819 (PTDC/OCE-ETA/27819/2017). It was also supported by National Funds through FCT—Foundation for Science and Technology under the Projects UIDB/05183/2020 [MED]; UIDB/00239/2020 [CEF]; CEECIND/02725/2018, UIDB/00100/2020, UIDP/00100/2020 [CQE]; UID/AGR/04129/2020, DL 57/2016/CP1382/CT0025 [LEAF].

References:

1. Canas S.; Caldeira I.; Belchior A.P.; Spranger M.I.; Clímaco M.C.; Bruno-de-Sousa R. Organisation of Vine and Wine. **2018**. Available online: http://www.oiv.int/en/technical-standards-anddocuments/collective-expertise/spirit-beverages (accessed on 15 April 2021).

2. Canas S., Caldeira I., Fernandes T.A., Anjos O., Belchior A.P., Catarino S. Improving Sustainable Viticulture and Winemaking Practices, Academic Press, Massachusetts, EUA, **2022**.

3. Granja-Soares J., Roque R., Cabrita M.J., Anjos O., Belchior A.P., Caldeira I., Canas S. Food Chem. (2020) 127450.