**Supplementary Material**

# Impact of cork closures on the volatile profile of sparkling wines during bottle aging

Filipa Amaro [1,2], Joana Almeida [1,2], Ana Sofia Oliveira [2], Isabel Furtado [2], Maria de Lourdes Bastos [1,2], Paula Guedes de Pinho [1,2,*], Joana Pinto [1,2,*]

[1] Associate Laboratory i4HB - Institute for Health and Bioeconomy, Department of Biological Sciences, Laboratory of Toxicology, Faculty of Pharmacy, University of Porto, 4050-313 Porto, Portugal

[2] UCIBIO/REQUIMTE, Department of Biological Sciences, Laboratory of Toxicology, Faculty of Pharmacy, University of Porto, 4050-313 Porto, Portugal

*Corresponding authors:
jipinto@ff.up.pt (J.P)
pguedes@ff.up.pt (P.G.P)

Address:
Associate Laboratory i4HB - Institute for Health and Bioeconomy
Laboratory of Toxicology, Department of Biological Sciences
Faculty of Pharmacy, University of Porto
Rua Jorge Viterbo Ferreira, 228
4050-313 Porto, Portugal
Tel.: +351 220428599; fax: +351 226093390

**Table S1.** Concentrations of other volatile organic compounds determined in sparkling wine #1, during bottle aging (12 to 42 months), for which no significant change was observed according to the type of closure.

| Class/Compound | 12 months [1] | | 24 months [1] | | 42 months [1] | | Units | Descriptors [2] |
|---|---|---|---|---|---|---|---|---|
| | 2D | MA | 2D | MA | 2D | MA | | |
| **Alcohols** | | | | | | | | |
| Isoamyl alcohol | 188 ± 34 | 157 ± 41 | 243 ± 7 | 252 ± 5 | 766 ± 68 | 573 ± 357 | mg/L | Banana, burnt cheese |
| 1-Octanol | 5.19 ± 0.08 | 4.79 ± 0.26 | 4.69 ± 0.30 | 4.48 ± 0.21 | 38.1 ± 2.04 | 34.3 ± 8.48 | µg/L | Rose, waxy |
| Phenylethyl alcohol | 53.9 ± 3.8 | 59.0 ± 12.9 | 19.3 ± 0.1 | 19.7 ± 2.2 | 38.7 ± 10.2 | 29.2 ± 11.4 | mg/L | Rose, honey |
| 1-Decanol | 33.2 ± 0.2 | 21.7 ± 15.4 | 36.9 ± 9.2 | 29.4 ± 5.0 | 26.2 ± 9.7 | 14.3 ± 10.3 | µg/L | Floral, fruity, waxy |
| **Aldehydes** | | | | | | | | |
| Benzaldehyde | 18.3 ± 1.9 | 19.1 ± 1.9 | 24.2 ± 2.1 | 24.2 ± 1.4 | 24.3 ± 6.8 | 17.2 ± 9.2 | µg/L | Almond, bitter, burnt sugar |
| Octanal | 0.24 ± 0.18 | ND | BLOQ | BLOQ | 0.78 ± 1.34 | 1.09 ± 1.44 | µg/L | Citrus, fatty |
| Phenylacetaldehyde | 13.8 ± 0.5 | 13.9 ± 0.3 | 35.5 ± 2.5 | 31.6 ± 0.8 | 86.9 ± 21.8 | 66.7 ± 21.2 | µg/L | Bitter, green, honey |
| Nonanal | 3.73 ± 1.78 | 1.61 ± 0.06 | 8.86 ± 4.61 | 6.68 ± 3.63 | 30.10 ± 34.2 | 36.3 ± 31.6 | µg/L | Green, pungent |
| Decanal | 8.21 ± 5.70 | 2.78 ± 1.48 | 3.68 ± 1.95 | 1.66 ± 0.47 | 1.69 ± 0.53 | 1.54 ± 0.91 | µg/L | Grassy, orange |
| β-Cyclocitral | 8.93 ± 0.02 | 8.92 ± 0.02 | 0.118 ± 0.204 | 0.147 ± 0.255 | ND | ND | µg/L | Floral, sweet |
| **Esters** | | | | | | | | |
| Ethyl 2-methylbutanoate | 6.55 ± 5.02 | 9.24 ± 4.25 | 14.4 ± 10.1 | 22.2 ± 6.8 | 30.0 ± 8.2 | 11.0 ± 8.9 | µg/L | Apple |
| Isoamyl acetate | 275 ± 189 | 376 ± 157 | 82 ± 42 | 132 ± 42 | 623 ± 142 | 284 ± 191 | µg/L | Banana, sweet, fruity |
| Hexyl acetate | 2.18 ± 0.05 | 1.90 ± 0.27 | BLOQ | BLOQ | 0.20 ± 0.17 | 0.11 ± 0.19 | µg/L | Cherry, pear |
| Ethyl hexanoate | 0.89 ± 0.15 | 0.69 ± 0.16 | 0.49 ± 0.29 | 0.74 ± 0.22 | 3.83 ± 1.60 | 1.60 ± 1.02 | mg/L | Apple, banana |
| Ethyl heptanoate | 3.25 ± 0.09 | 3.35 ± 0.06 | 0.31 ± 0.19 | 0.40 ± 0.25 | 2.73 ± 0.32 | 2.18 ± 0.45 | µg/L | Fruity, melon |
| Diethyl succinate | 14.7 ± 1.5 | 13.8 ± 1.5 | 27.9 ± 0.6 | 28.0 ± 2.1 | 88.7 ± 15.6 | 85.6 ± 6.9 | mg/L | Fruity |
| Ethyl octanoate | 1.86 ± 0.45 | 1.16 ± 0.37 | 0.49 ± 0.24 | 0.52 ± 0.15 | 5.43 ± 3.41 | 3.60 ± 1.55 | mg/L | Pineapple, sweet |
| Phenylethyl acetate | 25.4 ± 0.6 | 23.9 ± 1.0 | 6.2 ± 0.3 | 5.9 ± 0.3 | 4.4 ± 0.25 | 4.1 ± 0.8 | µg/L | Rose, violet |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Ethyl nonanoate | 9.5 ± 0.4 | 10.0 ± 0.8 | 3.5 ± 0.6 | 3.1 ± 0.8 | 33.6 ± 7.0 | 30.3 ± 10.2 | µg/L | Rose, fruity |
| Ethyl decanoate | 226 ± 22 | 204 ± 8 | 24.5 ± 7.9 | 13.1 ± 3.3 | 29.1 ± 20.4 | 31.7 ± 18.7 | µg/L | Brandy, fruity |
| **Furans** | | | | | | | | |
| Furfural | 0.85 ± 0.09 | 0.76 ± 0.04 | 1.49 ± 0.18 | 1.61 ± 0.11 | 3.70 ± 0.87 | 3.09 ± 2.14 | mg/L | Almond, bread |
| 5-Methyl-2-furfural | 43.1 ± 3.9 | 34.5 ± 3.4 | 28 ± 2.4 | 26.4 ± 1.7 | 42.6 ± 5.4 | 41.1 ± 11.2 | µg/L | Almond, burnt sugar |
| **Ketones** | | | | | | | | |
| 2-Heptanone | 0.26 ± 0.08 | 0.18 ± 0.07 | 1.93 ± 1.03 | 2.53 ± 0.51 | 5.53 ± 2.02 | 3.09 ± 2.22 | µg/L | Sweet, fruity, woody |
| 2-Nonanone | 1.75 ± 0.95 | 2.14 ± 1.05 | 3.72 ± 1.24 | 4.06 ± 0.73 | 3.23 ± 1.35 | 1.91 ± 1.64 | µg/L | Sweet, herbal |
| **Terpenes** | | | | | | | | |
| *α*-Pinene | BLOQ | BLOQ | ND | ND | ND | ND | - | Pine |
| 1,4-Cineole | 39.3 ± 28.6 | 64.7 ± 19.6 | BLOQ | BLOQ | ND | ND | ng/L | Minty, pine |
| Limonene | 3.03 ± 3.40 | 0.61 ± 0.20 | ND | ND | ND | ND | µg/L | Flowery, green, citrus |
| Eucalyptol | 0.90 ± 1.06 | 0.09 ± 0.07 | BLOQ | BLOQ | ND | 0.57 ± 0.99 | µg/L | Fresh, mint, eucalyptus |
| Linalool oxide | 238 ± 238 | 66 ± 12 | 48 ± 16 | 42 ± 8 | 188 ± 132 | 128 ± 52 | µg/L | Citrus, green |
| β-Linalool | 21.8 ± 4.4 | 18.4 ± 0.2 | ND | ND | ND | ND | µg/L | Flower, muscat, lavender |
| **Norisoprenoids** | | | | | | | | |
| α-Ionone | ND | ND | 1.34 ± 0.1 | 1.20 ± 0.1 | 2.4 ± 0.3 | 2.2 ± 0.8 | µg/L | Fruity, violet |

[1] Average concentration and standard deviation of sparkling wine #1 sealed with a sparkling cork with two natural cork discs (2D) and a microagglomerated cork (MA). A *n*=3 per closure was considered at 12 and 42 months, and a *n*= 4 at 24 months. [2] Descriptors reported in references [21,22]. BLOQ – below limit of quantification, ND – not detected, Q – qualitative alteration.

**Table S2.** Concentrations of other volatile organic compounds determined in sparkling wine #2, during bottle aging (12 to 42 months), for which no significant change was observed according to the type of closure.

| Class/Compounds | 12 months [1] | | 24 months [1] | | 42 months [1] | | Units | Descriptors [2] |
|---|---|---|---|---|---|---|---|---|
| | 2D | MA | 2D | MA | 2D | MA | | |
| **Alcohols** | | | | | | | | |
| Isoamyl alcohol | 71 ± 12 | 63 ± 6 | 252 ± 7 | 249 ± 11 | 544 ± 315 | 489 ± 117 | mg/L | Banana, burnt fruit, cheese |
| 3-Hexen-1-ol | ND | ND | 22.8 ± 5.4 | 23.2 ± 2.2 | 18.7 ± 17.8 | 9.4 ± 16.2 | µg/L | Green, leafy |
| 1-Hexanol | 0.26 ± 0.01 | 0.27 ± 0.01 | 5.75 ± 0.30 | 5.53 ± 0.18 | 1.77 ± 0.99 | 1.56 ± 0.25 | mg/L | Green, fruity |
| 1-Octanol | 6.21 ± 0.41 | 6.43 ± 0.62 | 6.72 ± 0.67 | 6.39 ± 1.04 | 33.6 ± 20.5 | 34.7 ± 2.9 | µg/L | Rose, waxy |
| Phenylethyl alcohol | 70.7 ± 12.2 | 62.9 ± 5.9 | 17.9 ± 3.2 | 19.8 ± 2.1 | 35.3 ± 20.4 | 30.3 ± 10.5 | mg/L | Rose, honey |
| 1-Decanol | 35.4 ± 4.9 | 33.0 ± 0.3 | 44.1 ± 13.9 | 43.3 ± 11.4 | 9.81 ± 11.6 | 4.63 ± 6.8 | µg/L | Floral, fruity, waxy |
| **Aldehydes** | | | | | | | | |
| Benzaldehyde | 30.4 ± 1.5 | 32.0 ± 2.7 | 36.7 ± 4.3 | 43.9 ± 5.7 | 25.9 ± 17.4 | 24.6 ± 3.6 | µg/L | Almond, bitter, burnt sugar |
| Octanal | 107 ± 132 | 77 ± 154 | BLOQ | BLOQ | 4 ± 6 | BLOQ | ng/L | Citrus, fatty |
| Phenylacetaldehyde | 34.4 ± 1.3 | 33.9 ± 1.7 | 32.2 ± 8.1 | 38.9 ± 3.4 | 89.8 ± 52.6 | 75.2 ± 21.2 | µg/L | Bitter, green, honey |
| Nonanal | 3.69 ± 2.32 | 4.89 ± 3.86 | 7.65 ± 7.09 | 14.3 ± 8.0 | 6.66 ± 7.76 | 6.45 ± 11.2 | µg/L | Green, pungent |
| Decanal | 1.61 ± 0.47 | 1.79 ± 1.02 | 2.20 ± 1.63 | 3.45 ± 0.73 | 2.11 ± 0.51 | 1.84 ± 1.00 | µg/L | Grassy, orange |
| β-Cyclocitral | 8.95 ± 0.01 | 8.95 ± 0.01 | 0.32 ± 0.32 | 0.19 ± 0.33 | ND | ND | µg/L | Floral, sweet |
| **Esters** | | | | | | | | |
| Ethyl isobutanoate | 113 ± 27 | 104 ± 13 | 192 ± 35 | 161 ± 56 | 122 ± 76 | 100 ± 19 | µg/L | Fruity |
| Ethyl butanoate | 0.44 ± 0.10 | 0.48 ± 0.05 | 0.65 ± 0.09 | 0.61 ± 0.18 | 2.56 ± 1.49 | 2.26 ± 0.29 | mg/L | Fruity, sweet, apple |
| Ethyl 2-methylbutanoate | 26.1 ± 6.9 | 25.2 ± 2.0 | 51.4 ± 9.2 | 49.3 ± 11.0 | 32.2 ± 17.6 | 24.0 ± 8.6 | µg/L | Apple |
| Ethyl isovalerate | 43 ± 10 | 42 ± 5 | 174 ± 29 | 144 ± 49 | 705 ± 393 | 567 ± 85 | µg/L | Fruity, sweet, spice |
| Isoamyl acetate | 102 ± 22 | 101 ± 11 | 141 ± 20 | 118 ± 36 | 433 ± 265 | 323 ± 51 | µg/L | Banana, sweet, fruity |
| Hexyl acetate | 1.33 ± 0.07 | 1.38 ± 0.09 | BLOQ | BLOQ | 0.13 ± 0.23 | BLOQ | µg/L | Cherry, pear |
| Ethyl hexanoate | 1.16 ± 0.24 | 0.91 ± 0.39 | 1.43 ± 0.21 | 1.20 ± 0.40 | 3.80 ± 1.09 | 3.28 ± 0.56 | mg/L | Apple, banana |
| Ethyl heptanoate | 3.58 ± 0.11 | 3.53 ± 0.07 | 1.15 ± 0.19 | 0.84 ± 0.21 | 2.80 ± 0.65 | 2.82 ± 0.27 | µg/L | Fruity, melon |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Diethyl succinate | 27.6 ± 2.3 | 26.9 ± 1.5 | 33.5 ± 4.0 | 35.8 ± 1.8 | 104 ± 22.8 | 124 ± 5.7 | mg/L | Fruity |
| Phenylethyl acetate | 26.3 ± 1.2 | 26.6 ± 1.8 | 8.0 ± 0.7 | 8.0 ± 0.3 | 5.6 ± 1.7 | 5.3 ± 0.3 | µg/L | Rose, violet |
| Ethyl nonanoate | 6.0 ± 0.3 | 6.0 ± 0.5 | 2.0 ± 0.6 | 1.5 ± 0.4 | 22.6 ± 14.6 | 20.9 ± 3.2 | µg/L | Rose, fruity |
| **Furans** | | | | | | | | |
| Furfural | 1.77 ± 0.19 | 1.77 ± 0.24 | 4.12 ± 0.33 | 4.25 ± 0.18 | 4.62 ± 2.78 | 4.16 ± 0.99 | mg/L | Almond, bread |
| 5-Methyl-2-furfural | 110 ± 15 | 96 ± 16 | 52 ± 4 | 51 ± 2 | 52 ± 27 | 51 ± 2 | µg/L | Almond, burnt sugar |
| **Ketones** | | | | | | | | |
| 2-Heptanone | 0.34 ± 0.06 | 0.43 ± 0.10 | 2.05 ± 0.23 | 1.81 ± 0.44 | 3.36 ± 2.01 | 2.77 ± 1.80 | µg/L | Sweet, fruity, woody |
| 2-Nonanone | 1.99 ± 0.40 | 2.90 ± 0.77 | 3.51 ± 0.26 | 4.04 ± 1.40 | 1.01 ± 0.66 | 1.39 ± 0.35 | µg/L | Sweet, herbal |
| 2-Undecanone | 0.09 ± 0.04 | 0.06 ± 0.04 | BLOQ | BLOQ | 3.98 ± 1.01 | 1.15 ± 1.56 | µg/L | Waxy, fruity |
| **Terpenes** | | | | | | | | |
| *α*-Pinene | ND | ND | ND | ND | ND | ND | - | Pine |
| 1,4-Cineole | ND | ND | ND | ND | ND | ND | - | Minty, pine |
| Limonene | 507 ± 107 | 655 ± 158 | ND | ND | ND | ND | ng/L | Flowery, green, citrus |
| Eucalyptol | 55.7 ± 14.4 | 60.4 ± 41.8 | BLOQ | BLOQ | ND | 572 ± 991 | ng/L | Fresh, mint, eucalyptus |
| Linalool oxide | 49.3 ± 14.1 | 64.8 ± 17.7 | 33.2 ± 7.5 | 44.2 ± 20.3 | 179.0 ± 93.9 | 131.0 ± 55.7 | µg/L | Citrus, green |
| β-Linalool | 21.8 ± 4.4 | 18.4 ± 0.2 | ND | ND | ND | ND | µg/L | Flower, muscat, lavender |
| **Norisoprenoids** | | | | | | | | |
| β-Damascenone | 2.31 ± 0.03 | 2.31 ± 0.05 | 1.53 ± 0.13 | 1.58 ± 0.13 | 3.17 ± 0.67 | 2.82 ± 0.06 | µg/L | Woody, floral, herbal |
| α-Ionone | ND | ND | 1.01 ± 0.04 | 1.13 ± 0.11 | 1.45 ± 0.67 | 1.28 ± 0.07 | µg/L | Fruity, violet |

***1*** Average concentration and standard deviation of sparkling wine #2 sealed with sparkling cork stopper with two natural cork discs (2D) and a microagglomerated cork (MA). A *n*=5 per closure was considered at 12 months, a *n*=4 at 24 months, and a *n*=3 at 42 months. *2* Descriptors reported in references [21,22]. BLOQ – below limit of quantification, ND – not detected, Q – qualitative alteration.