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Recycling in the winery: subcritical water as an eco-friendly process for the valorization of white wine lees

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This work highlights the potential for valorization of white wine lees using a subcritical water (SW) extraction process, which is both efficient and environmentally friendly compared to conventional processes. The extract obtained under optimal conditions using subcritical water, rich in antioxidant compounds, has demonstrated its ability to slow down the oxidation of wine, comparable to that of commercial yeast derivatives. These results open up promising prospects for the oenological use of lees extracts as natural antioxidants.

Context

Global viticulture produces nearly 260 million hectoliters of wine each year, generating significant volumes of by-products, including wine lees, which represent 2 to 6 % of the volume vinified. These deposits from fermentation and aging contain biomass rich in organic compounds. Currently, their recovery is mainly based on distillation (recovery of ethanol and tartaric acid), but a large part remains under-exploited[1]. However, lees have great oenological potential: they contain mannoproteins, lipids, peptides, and natural antioxidants such as glutathione (GSH), which plays a key role in preserving aromas and providing oxidative protection for wines[2]. White wine lees, which have not yet been extensively studied, are a promising source of bioactive molecules. In line with the circular economy, their extraction using eco-friendly processes opens up new opportunities for their use in oenology and other sectors, such as agri-food, cosmetics, and pharmaceuticals.

Extraction methods and analyses

White wine lees from a Sauvignon (AOC Bordeaux 2021) were freeze-dried and homogenized before extraction. Several solvents were tested (water, water/ethanol, water/methanol, or water/acetonitrile mixtures, 50:50) at a solid/liquid ratio of 1 % and 3 hours of extraction, followed by centrifugation and freeze-drying. Subcritical water (SW) extraction was performed in a high-pressure reactor (750 mL) with 2.5 g of lees and 250 mL of water at 40 bar. The parameters studied were temperature (100–250°C), duration (15–60 min), and agitation (100–1000 rpm). The extracts obtained were analyzed for their antioxidant activity using various complementary methods: the DPPH test (evaluation of free radical scavenging capacity), the FRAP test (measurement of ferric reducing power), and OCR (Oxygen Consumption Rate, estimation of the capacity to directly consume oxygen). Their composition was determined by quantifying total phenolic compounds (TPC), total sulfhydryl compounds (TSH), and reduced glutathione (GSH) by LC-HRMS[3].

Extraction of white wine lees

Several extraction solvents were compared (ethanol, methanol, acetonitrile, water at room temperature and subcritical water at different temperatures). Antioxidant capacity analyses (DPPH assay) showed that subcritical water extraction (Figure 1A) produced extracts that were four times more active than the initial lees and twice as active as those obtained using organic solvents. Subsequently, experimental optimization (Figure 1B) identified extraction temperature as the key parameter modulating the antioxidant capacity of the extracts (DPPH, FRAP, TPC, and OCR tests). Using modeling tools, it was possible to define the optimal extraction conditions (240°C, 15 min, 550 rpm, 45 bar, solid/liquid ratio 1 %) to obtain extracts with the highest antioxidant capacity.

The quantification of potential antioxidant compounds in the extracts (polyphenols, glutathione, and sulfhydryl groups) was also developed (data not shown). A correlation between antioxidant capacity and total polyphenols (TPC) as well as- SH compounds was observed[4]. However, glutathione was not detected in the extracts obtained under optimized conditions, probably due to its thermal degradation during SW extraction. This absence suggests the presence of other unidentified sulfur compounds contributing to the antioxidant activity of the lees. Thus, molecules other than polyphenols and GSH appear to be involved, as already observed for yeast derivatives, although their nature remains unknown. A study of the correlation between the composition of lees and the antioxidant activity of their extracts indicates that this activity does not depend on the initial antioxidant potential of the lees, nor on their initial polyphenol or glutathione content, but is rather correlated with their protein and lipid content[4]. This suggests that these compounds play a major role in the antioxidant functionality of the extracts. They could be the source of new molecules released or formed by subcritical water above 200°C. These results confirm the strong potential of subcritical water as an innovative and effective process for recovering antioxidant compounds from wine lees.

Study of the environmental effects of white wine lees extraction

We conducted a life cycle assessment (LCA) to evaluate the environmental effects of the various extraction processes applied to white wine lees.

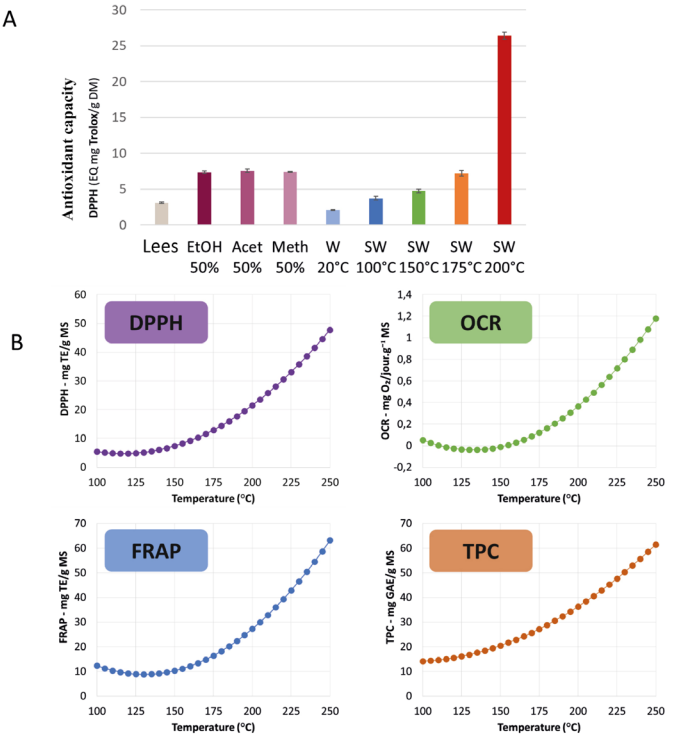


FIGURE 1. (A) Antioxidant capacity of the lees and their extracts obtained with different solvents. (B) Change in DPPH, FRAP, OCR, and polyphenol content (TPC) as a function of subcritical water (SCW) extraction temperature under constant operating conditions (RSL 1 % and 45 bar).

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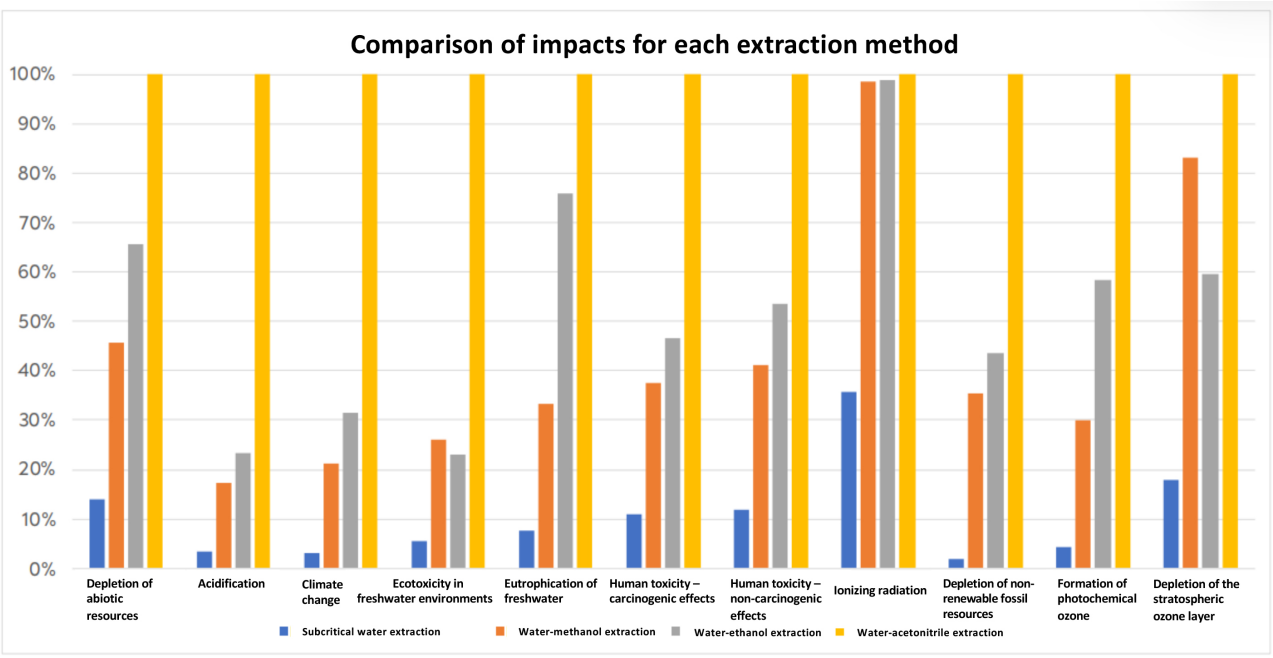


FIGURE 2. Life Cycle Assessment (LCA) of the extraction processes used for this study. LCA based on 11 conventional and standardized indicators (ISO 14040–44, EU Environmental Footprints). LCA carried out in collaboration with the company: Le Labo Durable.

Performed in accordance with ISO 14040 and 14044 standards, the LCA is based on experimental data collected at UMR Œnologie 1366 (ISVV, Bordeaux) for the extraction of 5 g of lees. Eleven environmental indicators were taken into account, including climate change, resource consumption, eutrophication, and human toxicity (Figure 2). The results show that the subcritical water process consistently has the lowest impact on all of the criteria evaluated, compared to methods using organic solvents. The main contributors to these effects are related to electricity consumption and equipment, while for solvents, they are production and the extraction operations themselves. Thus, in addition to its high extraction efficiency, subcritical water stands out for its favorable environmental profile, representing a sustainable and responsible alternative for the valorization of wine lees.

Oenological applications of antioxidant extracts from white wine lees

The extract with the highest antioxidant activity (in a model wine solution), obtained by subcritical water extraction under optimal conditions (SW at 240°C, 15 min, 45 bar, solid/liquid ratio 1 %), was evaluated in wine to study its potential for oenological applications. The extract was incorporated into a Colombard white wine, produced without the addition of SO_2, and then subjected to controlled oxidative conditions. To simulate successive oxygen additions during aging, the wine was subjected to three oxygenation cycles (saturation at 8.4 ± 0.4 mg O_2/L at 24°C) (Figure 3). At the same time, a comparison was made with commercial yeast derivatives, used as a reference for their recognized antioxidant properties[5]. The results showed that the lees extracts obtained by subcritical water extraction slow down oxygen consumption in wine, in a manner comparable to that observed with commercial yeast derivatives. We confirmed this trend for several wines and lees extracts. These observations suggest that the presence of antioxidant compounds in the extracts could help limit oxidation reactions in wine. This slowing down could be explained by the ability of these compounds to trap free radicals, thereby interrupting the oxidative chain reactions that occur in the absence of antioxidants. These observations confirm that subcritical water extraction increases the antiradical capacity of lees and allows the extraction or generation of active antioxidant compounds capable of limiting O_2 consumption in wine.

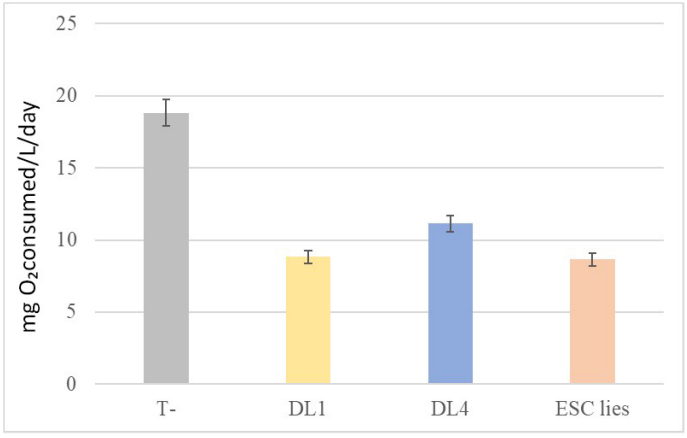


FIGURE 3. Oxygen consumption rate of wine after 3 saturations (8.4 mg/L of 02) for wine without antioxidants (T-), in the presence of yeast derivatives (DL1 and DL4 at 20 g/hL) and in the presence of subcritical water lees extract (SW lees at 20 g/hL).

Conclusion

Subcritical water proves to be very effective in extracting/forming antioxidant compounds from white wine lees. Tested in wine, these extracts reduce oxygen consumption, suggesting a potential benefit in limiting oxidation. These results open up prospects for the sustainable use of lees as a natural alternative to oenological additives. ■

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