

ABSTRACT

Wine maturation/aging is not just a winemaking technique but also an ancient tradition that has been used by numerous civilizations throughout history. Modern winemaking has continuously evolved through the adoption of various techniques and technologies aimed at improving wine quality. The modern winemaking process has progressed constantly through the use of various techniques and technologies to improve wine quality (Dündar, 2017). Wine maturation involves an extensive process, which sometimes contrasts with consumer demands and, consequently, incurs high costs. To mitigate these challenges, it is necessary to consider strategic interventions to accelerate winemaking processes while maintaining the required wine quality standards (Natolino and Celotti, 2022).

Following current trends in maturation process optimization, the doctoral thesis titled "Research on the Characteristics of White Wines from Iași Vineyard Obtained by Different Maturation Methods Using Oak Wood Fragments" aims to study the impact on the quality of white wines matured with oak wood fragments.

This research focuses on evaluating the effects of different maturation methods that can contribute to improving the quality of white wines, as well as optimizing the maturation process.

To achieve the overall research objective, the following specific objectives were established and accomplished: studying specialized literature and selecting a relevant database; establishing the experimental plan and conducting the tests according to the established methodology; determining and optimizing the optimal doses of oak wood-based products; analyzing the influence of maturation processes on the physicochemical properties of the wines obtained; monitoring the evolution of the main volatile and phenolic compounds during maturation; studying the influence of maturation procedures on the sensory changes in the wines; and interpreting and statistically analyzing the experimental results in relation to the claims in the specialized literature.

To achieve the established objectives, the basic physicochemical parameters of the obtained samples, the phenolic and volatile compound profile, and the sensory characteristics were analyzed. The data were statistically analyzed, and the effects of different ripening methods on the quality of the samples were monitored.

The administration of oak wood fragments generated statistically significant differences ($p < 0.05$) for most of the analyzed compounds. To achieve the specific objectives of this research, wines from Fetească regală and Sauvignon blanc varieties were obtained. The selection was based on their widespread distribution in the most representative vineyards in Romania, as well as their potential to interact favorably with the compounds released from the oak wood fragments. The grapes were harvested at full maturity, with a sugar concentration of 240 g/L, by hand, following optimal sanitary standards in 2021. The grapes were partially crushed and destemmed using a Delta E2 crusher-destemmer, and the resulting must underwent a 5-hour pre-fermentation maceration. Gentle crushing of the berries favored the release of aroma precursors and limited the transfer of chemical compounds responsible for undesirable sensory characteristics, such as astringency and herbaceous notes. After this process, the must was decanted and clarified by gravity for 24 hours.

The must was fermented in stainless steel tanks using yeasts from the *Saccharomyces cerevisiae* genus for the Sauvignon blanc, using Excellence® FTH product and for the Fetească regală the Excellence® B2 product, purchased from Lamothe-Abiet®. For both wines, the yeast dosage was 20 g/hL, according to the manufacturer's recommendations. Alcoholic fermentation took place at a controlled temperature of 14°C. After the alcoholic fermentation was completed, the wine was racked and divided in 5 L glass vessels, to which different doses of oak fragments (chips and granules) with three different degrees of toasting (*light*, *fresh*, and *medium*) were added for two periods of time, 10 and 20 days. For comparison, samples treated with the same doses and amounts of wood fragments were used, which were then subjected to ultrasound treatment for 15 minutes. The ultrasonic equipment used was a Bandelin Sanorex RK 1028

C model, which has a thermal power of 1450 W and an ultrasonic frequency of 35 kHz. Throughout the treatment, the temperature was controlled, maintaining a constant temperature of 20°C.

The oak wood fragments used in the experiment were from the species *Quercus robur* (French oak), used in the form of oak chips and granulars (*fresh*, *light*, and *medium*). To quantify the achievement of the proposed goal, the two types of oak wood fragments, each with three different degrees of toasting, were used to analyze the influence of heat treatments applied to the wood on the chemical composition of the wine and its organoleptic profile. Comparatively, ultrasound technology was also applied with the aim of intensifying the extraction of wood compounds and accelerating the maturation processes.

The wine samples obtained were stored under controlled conditions, at a temperature of 8 °C, with a relative humidity of 70-80%, and protected from light.

The results of the physicochemical analyses of the experimental variants fall within the limits permitted by the standards of the International Organization of Vine and Wine (OIV, 2020). Considering that all the samples were obtained from the same base wine, the observed differences between the variants are solely due to the applied treatments, namely the addition of oak wood fragments (in the form of granulars or chips), different dosages and contact times, as well as the use of ultrasound treatment, which accelerated the maturation processes.

In the case of the Sauvignon blanc samples, the total acidity varies within the range of 7.03-7.95 g/L tartaric acid (SVG12-SVG7US), with an increasing trend in the samples with higher wood dosages (2 g/L), especially in the *medium* -toasted chip variants, which favor a more intense extraction of phenolic compounds. Samples aged for 20 days (SVG7-SVG12 and SVG19-SVG24) show more pronounced trends of increasing total acidity, up to 7.80 g/L tartaric acid, and a slight decrease in pH, compared to the variants aged for 10 days. Increasing the oak wood dosage leads to an intensification of the volatile acidity of SVG13-SVG18 (0,38-0,39 g/L), an aspect associated with the additional oxygen supply and the amplification of the non-enzymatic oxidation process that occurs during maturation. The free and total SO₂ content slightly decreased in the ultrasound-treated variants, with the minimum concentration of free SO₂ recorded in sample SVG12US (40 mg/L), compared to the control sample SVG (48 mg/L).

In the case of samples obtained from the Fetească regală variety, the total acidity showed slight variations between samples, but there is a noticeable increasing trend in the variants matured with wood chips and those with 2 g/L FER13-FER19, compared to the control sample. Volatile acidity has the lowest value in the control sample and increases progressively with the intensification of the treatments, reaching the highest values in the samples with 2 g/L and a contact time of 10 days, especially in those matured with lightly or *medium* toasted chips (FER13-FER18). Additionally, the presence of oak chips can influence the chemical balance by releasing phenolic compounds, which can interact with wine components, modifying the pH and promoting the formation of volatile acids. Regarding SO₂, both total and free SO₂ showed decreasing trends, especially in the *medium* -roast and ultrasound-treated variants.

Phenolic compounds in wine can originate from grapes as raw material and from external sources, which include transfer from oak barrels, corks used for bottling, or various oenological treatments applied during the winemaking process. The analysis of phenolic compounds, performed on samples obtained from the Sauvignon blanc variety, reveals a slight decrease in the concentrations of catechin and epicatechin, depending on the wood treatment applied, a trend that corresponds to the behavior of flavonoids during traditional wine maturation. Syringic acid was identified in higher concentrations in the *medium* -toasted wood-matured variant, with a value of 0.12 µg/mL (SVG15), but was not detected in the control sample (SVG). Gallic acid varied depending on the type of wood used, with higher values in the variants matured with *fresh* wood chips and *light* roasted granules. The highest values of this compound are 4.29 µg/mL (SVG20) and 3.77 µg/mL (SVG22). Additionally, esculetin was also present in the ultrasound-treated variants, highlighting that cellular bonds are broken and minor phenolic compounds are extracted more efficiently. The detected value of esculetin in the analyzed samples was very low, 0.01 µg/mL (SVG8, SVG14). Analysis of the experimental data highlights significant variations in syringaldehyde concentration depending on the type of treatment

applied. The highest value was recorded in the SVG24 variant at 1.43 µg/mL, where *medium* -toasted chips were used at a dosage of 2 g/L for 20 days.

The highest caftaric acid values were recorded in SVG2US at 27.21 µg/mL, while the lowest value was determined in the SVG control sample at 24.80 µg/mL. This difference highlights the effectiveness of ultrasound treatment, which can contribute to better extraction of existing phenolic compounds by disrupting cell walls and increasing mass transfer.

In the case of samples obtained from the Fetească regală variety, epicatechin recorded the highest value in the control sample (FER), with a concentration of 0.67 µg/mL, and decreased significantly in the matured variants, 0.44 µg/mL in sample FER9. Gallic acid, which has the highest antioxidant potential among the compounds analyzed, showed a variation from 1.54 µg/mL in the control sample (FER) to 4.41 µg/mL in the FER20 variant, matured with *medium* -roasted granules at a dose of 2/L for 20 days. Syringaldehyde showed the highest concentration in the FER9 variant (1.44 µg/mL), while it was undetectable in the control sample, and caftaric acid recorded values between 36.31 µg/mL (FER) and 37.97 µg/mL (FER2US), indicating a small variation after the applied treatments.

Volatile compounds are essential for wine quality, directly determining its aromatic and taste profile. Volatile compounds present in the experimental variants were 7 alcohols, 13 esters, 15 volatile acids, and 33 other compounds (ketones, furanones, aldehydes, lactones, etc.).

In the analyses performed on Sauvignon blanc wines, the most relevant compounds in this class were 2,3-butanediol (both isomers), 1-butanol, 3-methyl (isobutanol), phenylethanol, and long-chain alcohols, such as substituted decanols and octanols. Isobutane, produced during amino acid metabolism, was present in all samples, with very similar variations between the two ripening treatments. However, higher concentrations are noted in samples matured with freshoak granulars (SVG1).

Ultrasound treatments did not lead to significant increases in higher alcohol concentrations, suggesting that their extraction is influenced by the type and dosage of wood used. Increasing the oak fragment dosage from 1 g/L to 2 g/L leads to a significant decrease in the isobutanol concentration, up to 57%. Relevant values were recorded for ethyl lactate, where a variation was observed from a minimum of 1.46% (SVG2) to a maximum of 3.61% (SVG1), a sample matured with *fresh* granulars at a dosage of 1 g/L for 10 days. Octanoic acid ethyl ester was detected in lower concentrations in most variants, reaching minimum values in the SVGUS sample. The preservation of volatile ester compounds in the analyzed wines is favored by short-term maturation (10 days), the use of low doses (1 g/L) with a low degree of toasting, and also the use of ultrasound as an emerging technological method. Acetic acid, the most common volatile acid in wine, recorded values between 2.68% (SVG2) and 7.93% (SVG1). This variation highlights the impact of maturation on the microbiological and biochemical processes that occur in wine. In the experimental variants, butyrolactone was detected in the range of 0.55% (SVG15) to 2.47% (SVG7). These values reflect an extraction following prolonged contact with the wood, which facilitates the degradation of lignin into volatile aromatic compounds. The long contact time, 10 and 20 days, allows for a progressive extraction.

Analysis of variance (ANOVA) applied to most compounds revealed significant differences between the samples ($p < 0.05$). For the samples that had prolonged contact with wood, the analysis revealed significant increases in the levels of lactone (VC9), diethyl succinate (VC12), and ethyl hydrogen succinate (VC17), which were associated with more intense notes of vanilla, ripe fruit, and aromatic complexity. Samples treated with ultrasound showed higher levels of isobutanol (VC1) and a reduction in acetic acid (VC6), which supports the preservation of freshness and fruity notes.

For the Fetească regală variety, the addition of wood fragments, as well as ultrasound, led to changes in the structure of the volatile compounds. The analysis revealed the presence of isobutanol (1-butanol, 3-methyl) in all samples, with concentrations ranging from 3.15% (FER3US) to 50.21% (FER9), suggesting that classic accelerated maturation can favor the accumulation of this compound. Esters, such as ethyl lactate showed concentrations between 0.70% (FER23) and 3.73% (FER8), with a slight increase observed in the ultrasound-treated samples. The dominant compound identified was butyrolactone, with values ranging from 0.60% (FER23) to 1.62% (FER16).

From the analysis of the LS (Figure 6.4) mean graph for the main volatile compounds of wines obtained from the Fetească regală variety, clear differences are evident between the samples aged only with the addition of oak wood and those subjected to ultrasound. The major peaks observed for VC1 (1-butanol, 3-methyl) and VC12 (diethyl succinate) in the samples treated for 10 and 20 days favored the accumulation of these compounds. In contrast, in the samples treated with ultrasound, the amplitude of these peaks is significantly reduced, indicating incomplete extraction due to the short contact time (15 minutes). Additionally, compounds such as VC7 and VC17 show higher concentrations in the variants matured-with the addition of wood fragments, due to their progressive release following prolonged interaction of the wine with the wood. In ultrasonic tests, the concentration distribution is more uniform and dominated by moderate values, with a preferential selection for compounds with a high diffusion coefficient and favorable solubility polarity being noted.

Sensory analysis of the samples obtained from the Sauvignon blanc variety highlights how the type of wood (chips or granulars), the degree of toasting (*fresh*, *light* and *medium*), the dosage (1 g/L, 2 g/L), and the application of ultrasound influenced the aromatic expression and taste balance of the wines. A comparative analysis reveals that the samples administered with granulars showed a reduction in vegetal and mineral notes compared to the control sample, but an increase in texture and persistence. These results suggest a relatively rapid transfer of aromatic compounds, with a decrease in vegetal notes.

Additionally, the samples where oak chips were used, had a more balanced profile, with better-integrated citrus and floral notes, maintained minerality, and a harmonious integration of vanilla notes. These characteristics indicate a gradual transfer of compounds from the wood. Ultrasound treatment resulted in a slight decrease in the intensity of the vegetal notes while preserving the freshness of the fruity aromas (elderflower, ripe fruit), which can be correlated with the short contact time and the limitation of the extraction of compounds responsible for the woody and lactonic notes. Samples matured with oak chips generated a more balanced and rounded aromatic profile. The intensities of the vegetal notes are reduced, and the moderate presence of vanilla and caramel notes reflects a controlled release of woody volatile compounds, while the high texture and persistence confirm the positive contribution of phenolic components to the taste structure.

From a sensory perspective, the aromatic profile of the Sauvignon blanc wines, specifically the witness sample (SVG), fully retained its varietal character, featuring green, citrus, and mineral notes. Maturation with fresh fragments (SVG1, SVG4) brought more intense aromas of elderflower and exotic fruits, while SVG1US presented the most pronounced vanilla note, thanks to rapid ultrasound extraction. At a dosage of 2 g/L, SVG13 and SVG22 offered a more structured and persistent profile, while SVG10US combined freshness with intense vanilla. For *medium* toasted wood, SVG6 and SVG12 balanced the woody aromas with the varietal ones, with SVG3 standing out for its coffee note, while SVG12US and SVG9US rapidly extracted surface compounds but with less developed roasting notes.

Sensory analysis of Fetească regală samples highlights that the addition of oak wood fragments, at different toasting levels and dosages, as well as the application of ultrasound, significantly influences the aromatic expression and taste balance. The witness samples (FER, FERUS) presented a young, vegetal profile with high acidity. The addition of *fresh* wood (FER8, FER11) reduced the vegetal notes and accentuated the fruity and floral aromas, while the dosage of 2 g/L, especially in combination with ultrasound (FER8US, FER11US), intensified the exotic fruit descriptors, taste persistence, and aromatic balance. The *light* wood offered a fresher and more harmonious profile, with notes of spice and coconut, while the *medium* wood (FER6, FER21) brought increased complexity through vanilla, tobacco, and coffee, but with a slight reduction in aromatic vivacity.

Due to the study investigating the use of ultrasound treatment and oak wood fragments to accelerate the maturation of white wine, the results demonstrated that these methods can reduce economic and environmental impacts while maintaining the sensory and physicochemical quality of white wines.