POLITECNICO MILANO 1863

SCUOLA DI INGEGNERIA INDUSTRIALE E DELL'INFORMAZIONE

Executive Summary of the Thesis

Accelerated Whisky Aging: Laboratory-Scale Process Optimisation

Laurea Magistrale in Food Engineering - Ingegneria Alimentare

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1. Introduction

Maturation is a key stage in whisky production, as it is during this period that the chemical composition of the spirit evolves, leading to the development of its characteristic aroma and flavor. Despite the global popularity and commercial significance of whisky, traditional ageing remains a time-intensive and costly process. These constraints have motivated continued research efforts aimed at improving and optimizing maturation practices [1].

Among these acceleration approaches, ultrasound-assisted techniques have been investigated for their ability to enhance extraction processes and promote the formation of flavor-relevant compounds [2]. Particular emphasis has been placed on lignin-derived congeners, including phenolic aldehydes such as vanillin, syringaldehyde, and coniferaldehyde, as well as related phenolic acids, which play an important role in defining the sensory profile of whisky [3].

This thesis aims to optimize the extraction of wood-derived congeners through the implementation of an improved experimental configuration, and to evaluate the influence of wood charring and toasting on the final congener concentrations.

2. State of the Art in Aging of Alcoholic Beverages

To examine recent developments in this field, a systematic review of studies published between 2023 and 2026 was conducted, focusing on the techniques and their effects on the chemical and sensory properties of alcoholic beverages. The insights and advancements identified in the literature were subsequently considered in the design of the present experimental work like the best treatment condition for toasting. The review further revealed that the majority of studies have primarily focused on wine aging, whereas comparatively fewer investigations have addressed aging acceleration in whisky. Table 1 summarized the studied alcoholic beverages and the methods used to control the aging process.

Alcoholic beverages and aging analysis techniques

Beverage	Methods
Whisky	Barrel aging; Wood fragments; Microwave radiation
Wine	Barrel aging; Underwater aging; Wood fragments; Pulsed electric field; Ultrasound; Bottle permeability to oxygen; Micro-oxygenation; Temperature modification; High hydrostatic pressure
Brandy	Wood fragments; Storage temperature modification; Ultra-high pressure
Distillate spirit	Ultrasound; Wood fragments; Rapid solid–liquid dynamic extraction
Fruit liqueur	Ultrasound; Wood fragments
Baijiu	Irradiation; Immobilised lipase; Electric field; Metal ions
Cachaça	Barrel aging; Wood fragments
Asake	Temperature control
Tequila	Wood fragments
Beer	Wood fragments; High pressure; Barrel aging
Champagne	Bottle permeability control
Cider	Fermentation modification
Astragalus mead	Ultrasound

Table 1: Summary of scientific studies published between 2023 and 2026 examining various alcoholic beverages, with emphasis on analytical methodologies for assessing aging characteristics and innovative techniques for monitoring and accelerating maturation processes.

3. Materials and Methods

3.1. Experimental methodology

The experimental framework of this study was based on a previous thesis investigating whisky ageing acceleration [4]. Several modifications were introduced to optimize the process and data acquisition. Temperature control was improved through continuous water circulation, allowing the system temperature to be maintained below 25 °C and enabling prolonged sonication without the need for intermittent on:off cycles previously required to prevent overheating.
Additional optimizations included the incorporation of a recirculation pump and a metallic reactor, enabling forced convection through continuous ethanol flow within the system. This configuration enhanced mass transfer and facilitated the extraction of wood-derived compounds. Ethanol solution 63% (v/v) was used instead of moonshine to minimize variability, and wood chips were selected instead of wood cubes to increase surface area to ethanol volume ratio and improve extraction efficiency.The final set up configuration is shown in Figure 1.

Figure 1: Final set up

A total of 70 wood chips of approximately regular geometry from oak wood were prepared, and their surface area was carefully measured for each experiment. Seven experimental configurations were designed following a one-variable-at-a-time approach to systematically isolate and assess the effect of individual parameters on whisky ageing acceleration (Table 2).

Experimental Conditions

Code	Wood treatment	Sonication
NN1	No wood	Yes
NN2	No pre-treatment	Yes
NN3	No pre-treatment	No
NN4	Toasted (180 °C, 20 min)	Yes
NN5	Toasted (180 °C, 20 min)	No
NN6	Charring	Yes
NN7	Charring	No

Table 2: Experimental conditions of the analysed samples.

3.2. Analysis methodology

High-performance liquid chromatography (HPLC) analyses were performed using a Restek C18 Ultra AQ column (5 µm particle size). Chromatographic separations were conducted at room temperature with a mobile phase flow rate of 1.1 mL/min. The method employed a ternary mobile phase system composed of acetonitrile, an aqueous solution of water and acetic acid (98:2, v/v), and a methanol–water–acetic acid mixture (70:29:1, v/v/v). Eluents volumetric flow rate during HPLC analysis is depicted in Figure 2.

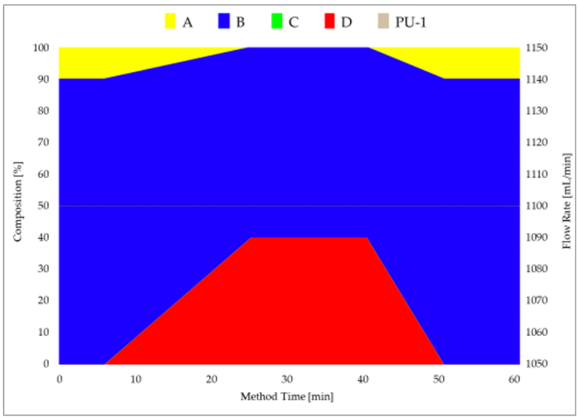

Figure 2: Eluents volumetric flow rate during HPLC analysis

Calibration standards shown in Table 3 were prepared in a 63% (v/v) ethanol matrix at defined concentrations and used to establish the analytical calibration model.

Calibration Standards

Calibration standard	concentration (ppm)
Vanillin	1, 5, 10
Vanillic acid	1, 5, 10
Syringic acid	1, 5, 10
Coniferaldehyde	1, 5, 10
Syringaldehyde	1, 5, 10

Table 3: Prepared concentrations for calibration standards.

For each of the seven experimental conditions shown in Table 2, 13 samples collected at different times (Table 4), filtered and stored in refrigerated conditions until analysis by high-performance liquid chromatography (HPLC).

Sampling Times

Sampling number	Sampling time
t0	0 min
t1	10 min
t2	20 min
t3	30 min
t4	1 hour
t5	1.5 hours
t6	2 hours
t7	2.5 hours
t8	3 hours
t9	4 hours
t10	5 hours
t11	6 hours
t12	7 hours
t13	8 hours

Table 4: Sampling times corresponding to each sampling number.

4. Results and Discussion

4.1. Water circulation

To control temperature during sonication, a continuous flow of tap water was applied to the ultrasonic bath. The temperature was monitored throughout the process to evaluate the effectiveness of the cooling system. Temperature data over 90 minutes of sonication showed that with continuous water circulation, the system temperature remained stable at 21–22 °C, enabling uninterrupted sonication without overheating or congener degradation. Without cooling, temperature rose to 35 °C in 30 minutes. As a result, the application of intermittent sonication cycles (on:off) to prevent overheating was no longer necessary. Continuous sonication could therefore be maintained until the desired concentration of wood-derived compounds was achieved.

4.2. Lignin-congeners identification

The experimental setup was designed to evaluate the accelerated maturation system under controlled reactor conditions. Due to the defined wood surface area-to-solution volume ratio and sonication time, several congeners were present at low concentrations after 8 hours.
Quantitative analysis therefore focused on syringaldehyde and vanillin, the most detectable compounds. Syringaldehyde was reliably quan-

tified, while vanillin was detected but below accurate quantification limits (Figure 3). The highest syringaldehyde concentration was observed under wood charring combined with sonication (sample NN6), indicating that this configuration enhanced compound formation and extraction.

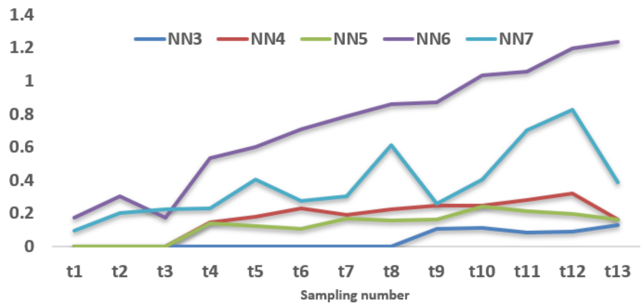

Figure 3: Syringaldehyde concentration in ppm over experiment

Figure 4 illustrates the progressive color evolution from the first to the last experimental sample, arranged from NN1 at the top to NN7 at the bottom. As shown, obvious gradual color development was observed in experimental runs involving wood pretreatment. The most intense coloration was recorded in the final sample corresponding to charred wood under sonication.

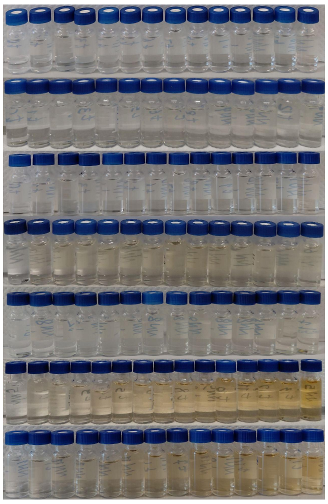

Figure 4: Samples from NN1 up to NN7

4.2.1 Analysis 1: Wood-Excluded Sonication

Figure 5 illustrates the chromatograms of sample NN1, conducted under sonication in the absence of wood pieces. The three chromatograms obtained at different sampling times exhibit nearly flat baselines, indicating no detectable formation of wood-derived compounds, indicating that ultrasound alone does not promote the formation of wood-derived maturation compounds.

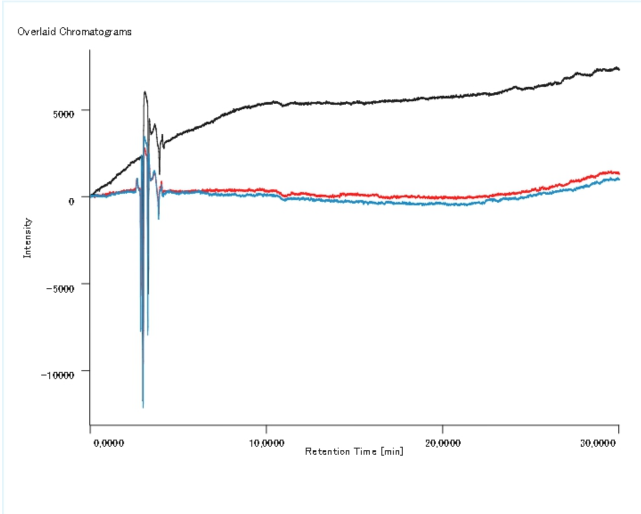

Figure 5: Blue line: Start of sonication, Red line: Midpoint sampling time, Black line: Final sampling time, the shift of black graph is because of detector drift, not a true chemical increase.

4.2.2 Analysis 2: No wood pretreatment

The experiment assessed the effect of sonication on untreated wood, serving as a reference for pretreated conditions. While slight color development occurred under sonication, chromatographic results showed limited influence on congener concentrations, with only syringaldehyde detected at later stages (Figure 6).

4.2.3 Analysis 3: Toasting

Toasted wood chips were evaluated under sonicated and non-sonicated conditions. Sonication promoted gradual color development and higher chromatographic peak responses in NN4 compared with NN5. Syringaldehyde concentrations increased over time in NN4 before slightly declining at the final sampling, while vanillin remained below quantification limits. Overall, ultrasound enhanced extraction efficiency under toasted wood conditions (Figure 7).

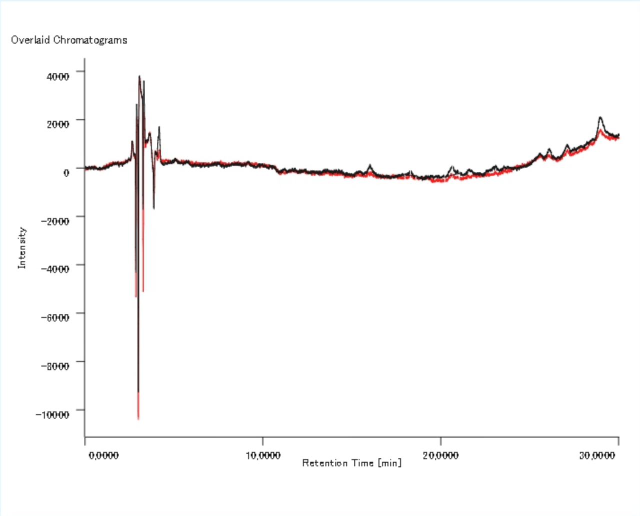

Figure 6: Red line: no pretreatment/sonication, black line: no pretreatment/no sonication

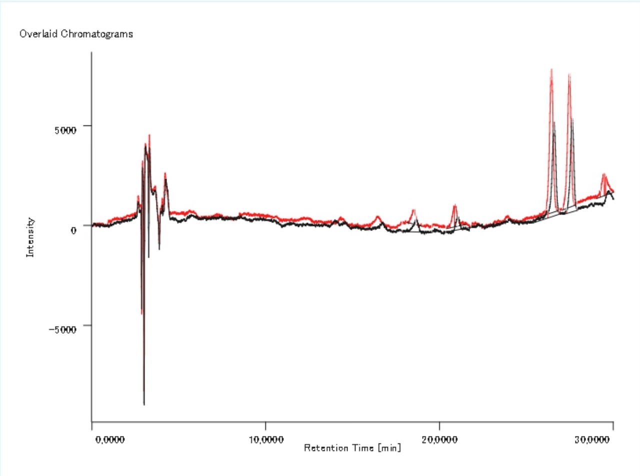

Figure 7: Red line: toasted/sonication, Black line: toasted/no sonication

4.2.4 Analysis 4: Charring

Charring experiments conducted with and without sonication demonstrated intensified color development and higher chromatographic responses in NN6. Syringaldehyde concentrations were greater under sonication, whereas vanillin remained below quantification limits (Figure 8).

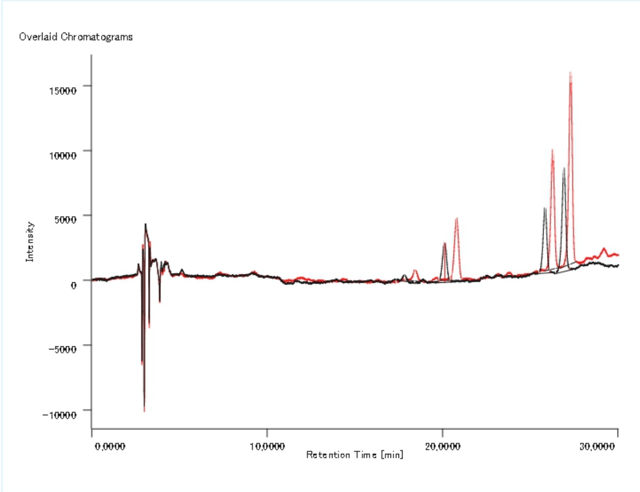

Figure 8: Red line: charred/sonication, Black line: charred/no sonication

4.2.5 Analysis 5: Wood pretreatment

At the final sampling point, comparison of chromatograms obtained under identical sonication conditions showed distinct differences among untreated, toasted, and charred wood samples. The charred wood exhibited the highest peak intensities, followed by toasted wood, while the untreated sample showed minimal peak development. These findings suggest that charring was the most effective pretreatment among the tested conditions, as reflected by both the greater number of detectable compounds and the higher chromatographic responses (Figure 9).

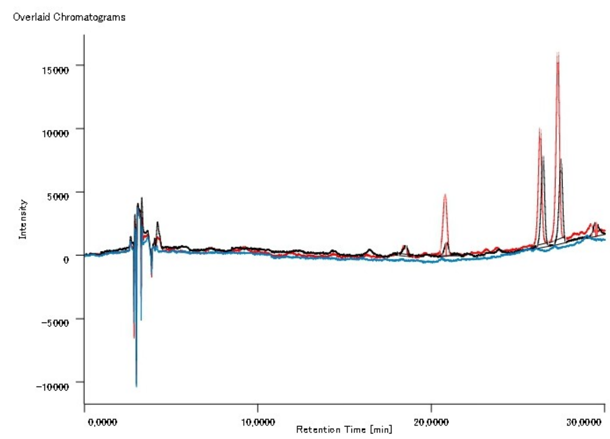

Figure 9: Red line: charred/sonication, Black line: toasted/sonication, and Blue line: no treatment/sonication

4.2.6 Analysis 6: Experiment time

Compared with the previous thesis, where the optimal sample (M-300) reached 6.5 ppm of syringaldehyde after a total of 100.5 hours, the corresponding charred wood configuration in this study (NN6) achieved 1.2 ppm within only 8 hours. Although the final concentration was lower, the substantial reduction in processing time indicates enhanced early-stage extraction kinetics. These results suggest that the modified setup effectively promotes initial congener release, demonstrating progress toward time-efficient maturation.

5. Conclusions

The modified experimental configuration, incorporating temperature control, a reactor, and forced circulation, enabled controlled ultrasound-assisted whisky maturation and supported measurable early-stage congener extraction within reduced processing times. Although absolute concentrations were limited by system and analytical constraints, the results demonstrate the potential of the setup for accelerating wood-derived compound formation.

Future work should focus on more sensitive analytical techniques, expanded compound profiling, optimization of wood pretreatment variables, evaluation of pulsed ultrasound, process scale-up, and sensory validation to assess industrial and commercial feasibility.

6. Bibliography

References

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[3] Thomas J. Kelly, Christine O'Connor, and Kieran N. Kilcawley. Sources of volatile aromatic congeners in whiskey. *Beverages*, 9(3), 2023.

[4] Master's thesis.

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