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STUDY ON THE IMPACT OF ULTRASOUND ON THE AGING PROCESS OF TRADITIONAL VIETNAMESE LIQUOR

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ABSTRACT

This study investigated the effects of ultrasound-assisted aging on the quality of traditional Vietnamese fruit and grain wines, specifically plum wine and purple sticky rice wine. Using a 5 mm diameter ultrasound probe at amplitudes of 20% and 25% for durations of 5, 10, and 15 minutes, the changes in methanol content, total polyphenol content, total monomeric anthocyanin content, and sensory characteristics were evaluated. Results showed that ultrasound treatment significantly reduced methanol levels across all samples, with reductions up to 40 - 70% depending on the wine type and treatment duration. Polyphenol and monomeric anthocyanin content show a decrease of 20-80% through the treatments. They exhibited a two-phase trend: an initial increase due to enhanced cell disruption and extraction, followed by a decline likely caused by oxidation under prolonged or high-intensity exposure. Sensory analysis indicated that mild ultrasound conditions (e.g. 20% amplitude for 5 minutes) improved aroma and reduced harshness, whereas higher intensity and longer durations led to unfavorable sensory changes. These findings align with previous studies–and suggest that controlled ultrasound application can enhance the aging process of Vietnamese traditional wines by improving quality while reducing harmful methanol content, heading to apply for industrial production.

Keywords: Ultrasound, aging process, plum wine, purple sticky rice, polyphenol, methanol level.

1. INTRODUCTION

Currently, ultrasound is considered a non-thermal method for application in the Food Technology industry, especially in liquid products. The cavitation phenomenon is generated by ultrasound waves, creating microbubbles and evenly distributed in the liquid. When the wave oscillation is large enough, these bubbles burst, causing energy accumulation and generating extremely high temperatures and pressures in the cavitation area [1]. This will change the physical, physicochemical, and chemical properties of the product. At the same time, ultrasound waves also have an impact on sensory properties, including smell, taste, and color. In general, the ultrasound method has a strong influence on the aging process of liquors [2].

In recent years, studies have mainly focused on the effects of ultrasound on red wine, white wine, and some fruit vinegars. Mainly, the phenolic compounds, volatiles, flavors, and colors of the subjects have been studied and analyzed [3], [4]. For Asian wines, studies have mainly focused on the fermentation process and the reuse of rice wine and rice vinegar yeast residues [5], [6]. For the aging process, He et al. evaluated the effects of ultrasound in a 20 L chamber and found that the contents of esters, aldehydes, volatile alcohols, and flavor compounds changed after the application of ultrasound [7]. However, the results obtained in these studies are still insufficient to draw out the ultrasound modes and methods using probes or ultrasound baths that can be applied to other traditional Asian wines.

Traditional Vietnamese wine is a typical Asian wine. With the common production method of using high alcohol wine and then aging it for a long time to extract beneficial compounds, a batch of wine needs a minimum of 6 months to reach an acceptable level. During the soaking and aging process, many reactions occur in the wine, including oxidation, esterification, reducing the "shock" taste, making the product easier to drink, and better for health. The traditional wine aging method has major disadvantages, including a long time, high cost, and difficulty in controlling stable quality for batches of products because it depends on many factors such as temperature, humidity, etc.

This study focuses on the effects of ultrasound waves on some typical indicators of Vietnamese craft wine and compares them with traditional wine aging methods, from which it is possible to choose a suitable ultrasound mode, aiming to apply it in stable industrial production.

2. MATERIALS AND METHODS

2.1. Materials

Wine samples were produced from Hanoi plum and purple sticky rice according to the process in Figures 1 and 2. Two wine samples were divided into 2 types: fresh wine that had just finished the fermentation process without aging time (0 day) and wine after aging for 1.5 years according to the traditional method. Wine samples were coded according to Table 1.

Table 1. Sample codes

Types of wine	Aging time	Codes
Plum wine	0 day	R1
Plum wine	1.5 years	R2
Purple sticky rice wine	0 day	R3
Purple sticky rice wine	1.5 years	R4

2.2. Methods

2.2.1. Ultrasound treatment

The QSONICA SONICATORS model CL-18 ultrasound device with a 5 mm diameter probe was used for the survey. The device provides ultrasound with a frequency of 20 kHz and a power of 125 W. 50 mL of wine samples were processed with the probe 1 cm from the bottom, with the applied mode of 2 seconds of running and 1 second of resting, and kept at a temperature of 17 ± 2 ºC to avoid the decomposition of alcohol-sensitive compounds.

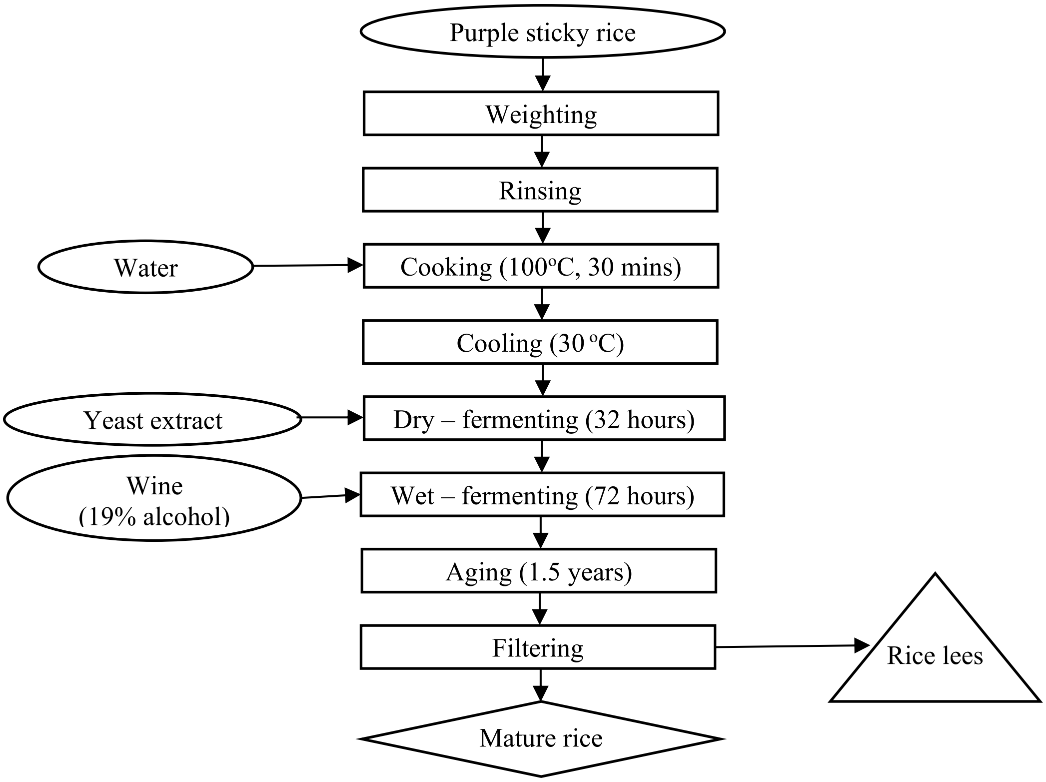

Figure 1 . Purple sticky rice wine preparation

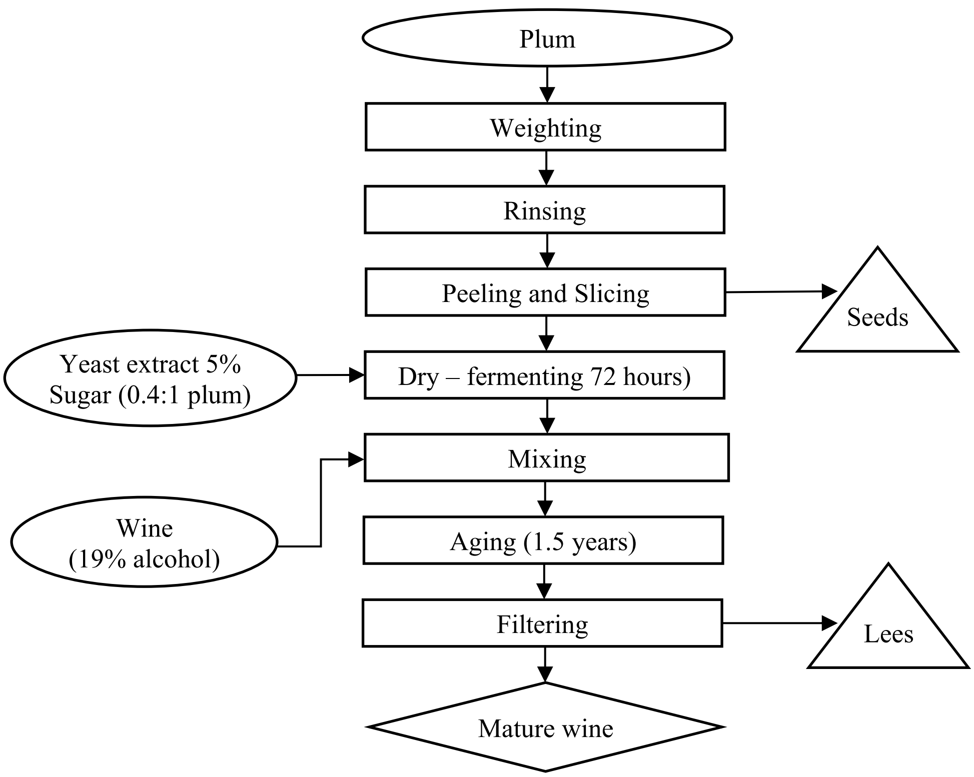

Figure 2. Plum wine preparation

Six modes were investigated in 3 repetitions in this study, which are a combination of two factors: time (5, 10, 15 minutes) and amplitude (20, 25%) [8]. After the ultrasound process, the wine samples will be stored in PE packaging at a temperature of – 40 °C for subsequent experiments.

2.2.2. Total polyphenol content analysis

Using the Folin–Ciocalteu micro method to determine the total phenolics (TPC) in wine [9]. Constructing the Folin–Ciocalteu Gallic Acid Standard Curve based on Absorbance Units to determine the total polyphenol content in mg/L gallic acid units.

2.2.3. Total monomeric anthocyanin pigment content analysis

Total monomeric anthocyanin pigment content (TMAPC) was determined based on AOAC 2005.02. Total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines. pH differential method [10].

2.2.4. Methanol content analysis

Methanol content was determined by the official methods outlined in the Vietnamese National Standard (TCVN 8010:2009) [11].

2.2.5. Sensory evaluations

Twelve semi-trained judges from the Chemical Engineering Faculty of Danang University of Science and Technology were selected to evaluate the overall acceptability of the samples (4 wines with 6 treatments). This evaluation is based on a 5-point Hedonic rating scale that shows the level of acceptability from 1 (disliked extremely) to 5 (liked significantly). The sensory acceptability points (SAP) were analysed by the ANOVA method [12].

2.2.6. Data analysis

The data obtained was designed using Design Expert version 13 software (Stat-Ease company, Minneapolis, Minnesota, USA). For each response variable, analysis of variance (ANOVA) was conducted to determine significant differences among the treatments.

3. RESULTS AND DISCUSSION

3.1. Total polyphenol content (TPC)

The changes in total polyphenol content after ultrasound treatment modes are shown in Figures 3 and 4.

During natural aging using traditional brewing methods, phenolic compounds in craft liquors are oxidized by more than 90% due to oxidation. The survey results showed that the impact of ultrasound on total polyphenol content (TPC) in liquor depends significantly on the amplitude and treatment time, and also varies according to each type of liquor sample. Specifically, with sample R1, when using an amplitude of 20%, TPC tends to decrease sharply over time, while at an amplitude of 25%, TPC increases slightly at 10 minutes and then decreases again when extended to 15 minutes. This shows that ultrasound at moderate amplitude and short duration can support the release of TPC in liquor, but if prolonged or used at high intensity, the oxidation process can reduce TPC content due to the decomposition of the phenolic structure. In sample R2, TPC decreased continuously from 5 to 15 minutes at both amplitudes, indicating that this liquor sample has an unstable TPC structure and is easily affected by ultrasound waves. In contrast, sample R3 had almost no obvious change in TPC, especially at 20% amplitude, indicating that polyphenols in this sample are more stable or less affected by ultrasound waves. Sample R4 recorded a significant decrease in TPC at 20% amplitude, while 25% amplitude slightly increased TPC for 10 minutes, then caused a sharp decrease at 15 minutes, demonstrating that the reaction characteristics of this sample are quite complex.

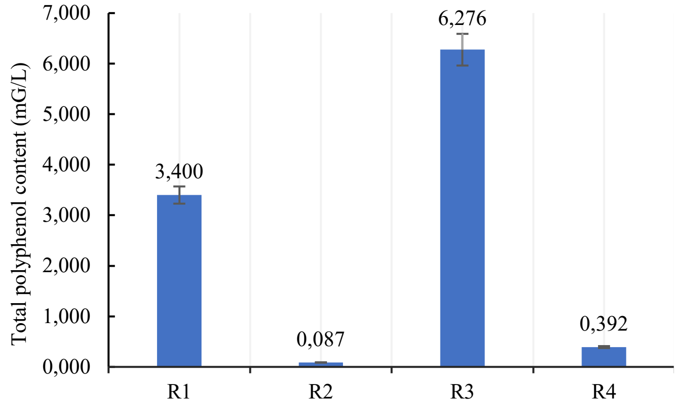

Figure 3 . Total polyphenol content of untreated samples

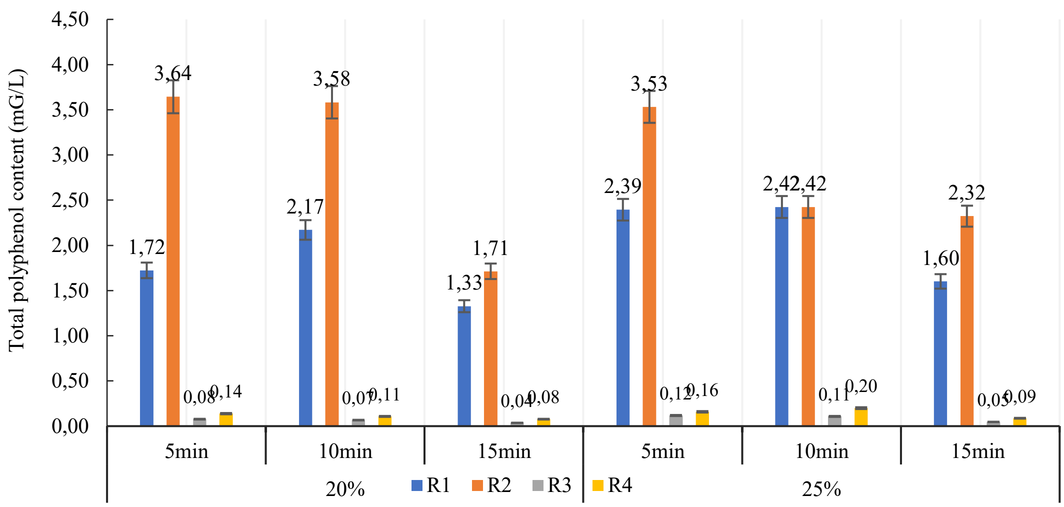

Figure 4. Total polyphenol content of treated samples

The ultrasound method could retain phenolic compounds in liquor more effectively than traditional liquor aging methods [13]. The change in phenolic composition may be due to polymerization, depolymerization, co-pigmentation, isomerization, and decomposition processes that occur during the ultrasound process [14]. Cavitation plays an important role in these reactions, causing oxidation of phenol to quinones and generating free radicals that degrade phenolic compounds. This suggests that ultrasound can promote phenolic depolymerization and recombination, and accelerate liquor aging reactions if applied under suitable conditions [15]. Compared with previous studies, this trend is completely consistent. Zhang et al. and Ferraretto et al. demonstrated that short-time and moderate-power ultrasound can promote the release of polyphenols, but if the time is prolonged or the power is increased too much, oxidation will cause TPC degradation[16], [17]. From this, it can be concluded that the optimal ultrasound condition for liquor aging to preserve TPC is 20% amplitude for 5 – 10 min, which is especially effective in samples R1 and R2. Extending the treatment time or increasing the intensity should be considered and further investigated for each liquor sample to avoid reducing the sensory quality and biological value of the product.

3.2. Total monomeric anthocyanin pigment content (TMAPC)

The changes in total monomeric anthocyanin pigment content after ultrasound treatment modes are shown in Figures 5 and 6.

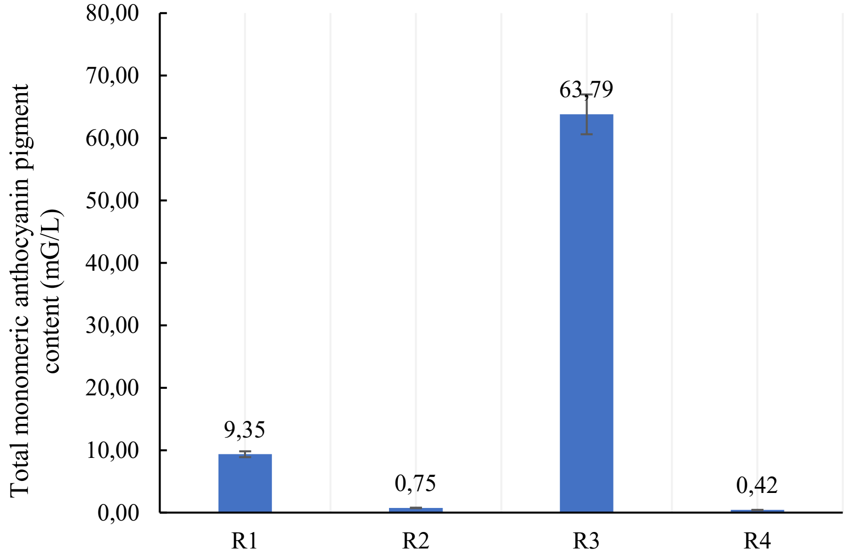

Figure 5. Total monomeric anthocyanin pigment content of untreated samples

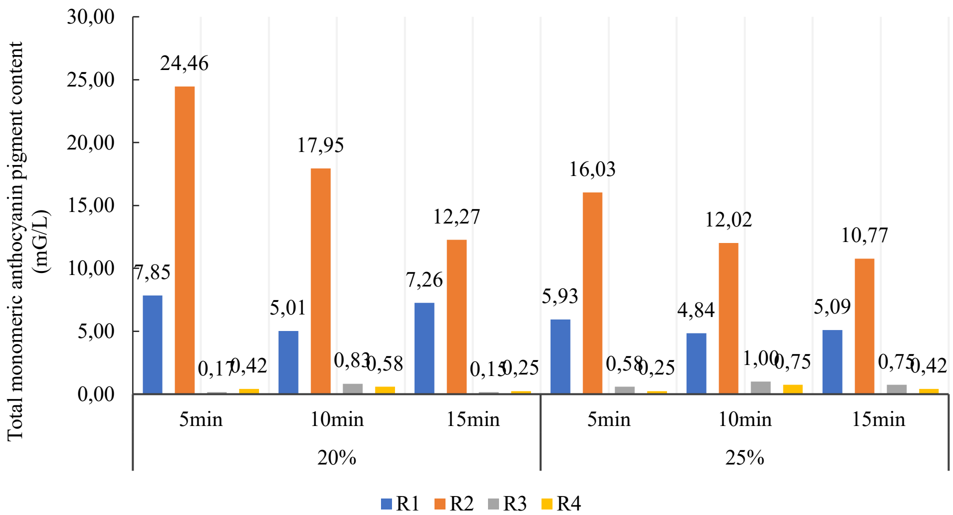

Figure 6. Total monomeric anthocyanin pigment content of treated samples

The anthocyanin content decreased significantly, by about 95%, when aging by the traditional aging method. The results showed that the ultrasound liquor treatment process had a significant effect on the total monomeric anthocyanin pigment content (TMAPC) in the liquor samples. In sample R1, when using ultrasound at 20% amplitude, the TMAPC content decreased slightly by about 6%, from 7.85 mg/L to 7.26 mg/L; meanwhile, when increasing the amplitude to 25%, TMAPC also decreased by only 10% after 15 minutes of treatment. For sample R2, the decrease at 20% amplitude was 12%, and even only 6% when using 25% amplitude, showing that, under certain conditions, ultrasound can help preserve anthocyanins better. In contrast, samples R3 and R4, which were aged for a long time, showed a very strong decline, with TMAPC remaining only about 5% of the initial value, and a nearly negligible decrease (about 2%) after ultrasound treatment at both 20% and 25% amplitudes. These results reflect the tendency of anthocyanin degradation under the influence of ultrasound, especially when the liquor has undergone a long oxidation process.

The aging process is very complex, which causes many reactions such as oxidation, degradation, polymerization, condensation, and structural changes, leading to changes in the color of liquors. It has been reported that significant effects could be induced by ultrasound cavitation on these aging processes, according to amplitude, frequency, and duration [18]. Especially, the condensation reaction between anthocyanins and tannins could be enhanced by ultrasound, and this effect potentially reduced the color evolution during the aging process [19]. Explained this, the acoustic cavitation can generate radical species in the water medium, which can enhance the polymerization and condensation reactions of chemical species [20]. Xie et al. noted that moderate-power ultrasound can improve liquor color stability by limiting anthocyanin degradation [21]. However, the use of high-power ultrasound or long treatment times promotes the degradation of anthocyanins and phenolic compounds in red liquor [22]. This degradation may be due to the degradation of anthocyanins, opening of the benzene ring, and formation of charcoal [18]. The light increase of the TMAPC in R3 can be explained [23]. Thus, the effect of ultrasound on anthocyanins is bidirectional: it can aid the extraction or preservation of anthocyanins in the early stages, but it also risks rapid degradation when the optimal amplitude and treatment time are exceeded.

3.3. Methanol content

The results of the semi-quantitative methanol content survey are shown in Figure 7.

(a) R1 (b) R2 (c) R3 (d) R4

Figure 7. Methanol content changes of 4 samples

After ultrasound treatment, the methanol content in all liquor samples showed a clear decrease, as shown by the color comparison results. For liquor R1 and R2 – two samples commonly used in the 5-, 10-, and 15-minute time surveys – the color intensity gradually decreased, indicating a significant decrease in methanol, especially at 15 minutes. In samples R3 and R4, although the change was not as strong, a gradual fading of the color intensity was still observed with treatment time, indicating that methanol also decreased. Overall, the results showed that treatment time and ultrasound conditions influenced the degree of methanol reduction, with estimated reductions ranging from about 40% to over 70% compared to the original sample, especially when the time was prolonged, and the ultrasound power was higher [24]. Chang et al. noted that after ultrasound treatment of rice liquor at 20 kHz frequency for different times, the methanol content showed a decrease from 58 to 54 mg/L [8]. At the same time, Hao et al. also mentioned the decrease of volatile compounds, including methanol, when using ultrasound to assist the rice fermentation process [5].

The cavitation phenomenon created by ultrasound can promote the oxidation of methanol to formaldehyde and further conversion to formic acid, significantly reducing the methanol content in liquor. This observation is also in agreement with the results reported by Gavahian et al.. The paper warns that uncontrolled ultrasound conditions may lead to the formation of harmful by-products. However, when properly controlled, ultrasound improves liquor quality and accelerates aging without producing dangerous levels of toxic compounds [25]. The same finding was pointed out by Chang, A. C., et al, that the methanol content was reduced after treatment by using an ultrasound bath, suggesting that ultrasound promotes the oxidation of methanol into compounds such as formaldehyde and formic acid [8], [26]. The study concluded that ultrasound could be a promising tool for simulating natural aging in a shorter time [27]. However, the reduction depends on specific conditions such as frequency, power, temperature, and processing time – factors that need to be optimized if effective methanol removal is desired while still ensuring the sensory quality of the liquor.

3.4. Sensory evaluations

Sensory evaluation results from 12 participants showed that the ultrasound treatment produced significant changes in the taste and aroma of the liquor [28]. Among the survey modes, the three conditions including 20% – 5 minutes (4.63 points), 20% – 10 minutes (4.55 points) and 25% – 5 minutes (4.70 points) all achieved the highest average scores, ranging from 4.5 to 4.7, indicating good sensory acceptance and few negative changes. In particular, 25% – 5 minutes was the mode with the highest score (4.70 points), indicating that a slight increase in amplitude but keeping the time short can improve the taste without causing adverse phenomena.

In contrast, when the time was prolonged, especially at 25% – 15 minutes, the sensory score decreased sharply to 2.45 ± 0.015, the lowest of all samples. This suggests that prolonged sonication at high intensities may result in the appearance of undesirable odors and flavors, such as sour, pungent or fermented odors [29]. The scores also dropped significantly at 25% – 10 min (3.36) and 20% – 15 min (3.25), reinforcing the trend that too long or too intense sonication causes negative sensory changes.

This phenomenon can be explained by the fact that the strong ultrasound process facilitates the evaporation of volatile compounds and the formation of organic acids, causing sour or pungent odors. Zhang et al. reported that total acids increased due to the oxidation of alcohols and aldehydes and the formation of acids in liquors [30]. The enhanced esterification effect by the reaction between alcohols and acids has also been indicated by Ince et al. [31].

In addition, there may be metabolic products from methanol, aldehydes, or protein degradation products from microorganisms under the stimulation of ultrasound, directly affecting the sensory properties of the liquor product.

Table 2. Influence of ultrasound treatments on the sensory acceptability of liquors

Samples	R1	R2	R3	R4
20% - 5 mins	4.63 ± 0.035	4.55 ± 0.027	4.70 ± 0.015	4.51 ± 0.011
20% - 10 mins	4.68 ± 0.023	4.47 ± 0.015	4.88 ± 0.022	4.62 ± 0.016
20% - 15 mins	3.03 ± 0.041	3.83 ± 0.031	3.57 ± 0.034	2.89 ± 0.037
25% - 5 mins	4.76 ± 0.015	4.25 ± 0.021	4.80 ± 0.018	4.16 ± 0.015
25% - 10 mins	3.92 ± 0.032	3.24 ± 0.014	3.87 ± 0.016	4.13 ± 0.021
25% - 15 mins	3.54 ± 0.017	2.95 ± 0.031	3.08 ± 0.032	2.45 ± 0.015

3.5. Model equations for TPC, TMAPC, and SAP

3.5.1. Model selection

Selecting models for each investigated property of liquors is important to analyze the impact of ultrasound on the product and optimize the aging process using an ultrasound probe. There are 5 models used in this research, including Linear, Mean, Quadratic, and 2FI (Table 3). The sequential p-values (p<0,005) show the improvement of the chosen model, compared to simpler models. The Adjusted R^2 reflects how well the model explains the variation in the response variable, adjusting for the number of predictors, and provides a more accurate measure than R^2 when comparing models with different numbers of terms. Predicted R^2 assesses how well the model predicts new data, then a negative or very low value suggests that the model performs poorly in predicting unseen data.

Table 3. Fit Summary of TP of the outputs of the samples

Sample	Output	Sequential p-value	Adjusted R^2	Predicted R^2	Model
R1	TPC	0,0001			Mean
	TMAPC	0,0160	0,7567	0,4014	Quandratic
	SAP	0,0002	0,8590	0,7951	Linear
R2	TPC	0,1296	0,2500	-0,1496	Linear
	TMAPC	0,0026			Mean
	SAP	0.0114	0.592	0.3289	Linear
R3	TPC	0.0402	0.4404	0.0788	Linear
	TMAPC	0.0169	0.5494	0.4033	Linear
	SAP	0.0006	0.8020	0.6733	Linear
R4	TPC	0.0090	0.6151	0.3996	Linear
	TMAPC	0.0447	0.3110	-0.7718	2FI
	SAP	0.0015	0.7547	0.6276	Linear

3.5.2. Model equations of sample R1

$$Model_{TPC} = +0.096100 - 0.004164X_{duration} + 0.000772X_{amplitude}$$

$$Model_{TMAPC} = +0.420909 - 0.000351X_{duration} + 0.000417X_{amplitude}$$

$$Model_{SAP} = +8.54926 - 0.084105X_{duration} - 0.182223X_{amplitude}$$

3.5.3. Sample R2

$$Model_{TPC} = +1.92909 + 0.000496X_{duration} + 0.000384X_{amplitude}$$

$$Model_{TMAPC} = +41.67936 - 1.02105X_{duration} - 2.54808X_{amplitude} - 0.005X_{duration}X_{amplitude} + 0.051567X^2_{duration} + 0.052415X^2_{amplitude}$$

$$Model_{SAP} = +6.27872 - 0.155425X_{duration} - 0.035714X_{amplitude}$$

3.5.4. Sample R3

$$Model_{TPC} = +6.97259 - 0.150056X_{duration} - 0.121373X_{amplitude}$$

$$Model_{TMAPC} = +61.00797 - 0.976525X_{duration} - 1.73119X_{amplitude}$$

$Model_{SAP} = +5.59764 - 0.108355X_{duration} - 0.022566X_{amplitude}$

3.5.5. Sample R4

$Model_{TPC} = +0.027932 - 0.010321X_{duration} + 0.009891X_{amplitude}$

$Model_{TMAPC} = +3.74333 - 0.293604X_{duration} - 0.150405X_{amplitude} + 0.0134X_{duration}X_{amplitude}$

$Model_{SAP} = +6.26237 - 0.134179X_{duration} - 0.051056X_{amplitude}$

3.6. Optimization of the aging process using the ultrasound probe

The optimization of process variables, including duration and amplitude, is necessary to get the maximum output of the product. Accordingly, the predicted values indicated in Table 4 using Design Expert 13 of desired conditions with 20% amplitude in 5 minutes processing, resulted in an optimum value of the total polyphenols content, total monomeric anthocyanin content, and sensory acceptability to the 4 samples of liquors.

Table 4. Predicted properties of liquors treated by optimal ultrasound-aging process with 20% - amplitude in 5 minutes

Sample	Total polyphenols (mg/L)	Total monomeric anthocyanin pigment content (mg/L)	Sensory acceptability point	Desirability
R1	1.929	7.368	4.787	0.864
R2	0.091	0.422	4.484	0.657
R3	3.795	21.502	4.605	0.789
R4	0.174	0.607	4.570	0.763

4. CONCLUSION

The results of the study showed that the application of ultrasound had a clear impact on the sensory quality and chemical composition of Vietnamese craft liquors, specifically plum liquor and sticky rice liquor. The content of polyphenols and anthocyanins in liquor tended to decrease with increasing amplitude and prolonging the ultrasound time, especially at 25% - 15 mins, indicating significant oxidation. However, when treated under optimal conditions (20% for 5 minutes), the liquor still maintained appropriate levels of polyphenols and anthocyanins without too much degradation, while improving the sensory property with a new, more pleasant taste. The methanol content in liquor samples also decreased significantly, especially in aged plum liquor, showing the potential of ultrasound in reducing food safety risks. However, the 0-day sticky rice liquor sample did not record a clear change in methanol, due to its low methanol content from the beginning. Overall, ultrasound under appropriate controlled conditions not only improves safety but also contributes to the creation of liquor products with novel sensory qualities, with potential applications in craft liquor production.

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TÓM TẮT

NGHIÊN CỨU ẢNH HƯỞNG CỦA SÓNG SIÊU ÂM ĐẾN QUÁ TRÌNH LÃO HÓA CỦA RƯỢU TRUYỀN THỐNG VIỆT NAM

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Nghiên cứu này khảo sát tác động của quá trình lão hóa hỗ trợ bằng sóng siêu âm đến chất lượng của các loại rượu truyền thống Việt Nam làm từ trái cây và ngũ cốc, cụ thể là rượu mận và rượu nếp cẩm. Sử dụng đầu dò siêu âm đường kính 5 mm ở biên độ 20% và 25% trong các khoảng thời gian 5, 10 và 15 phút, các thay đổi về hàm lượng methanol, tổng polyphenol, tổng anthocyanin dạng monomer và đặc tính cảm quan đã được đánh giá. Kết quả cho thấy xử lý siêu âm làm giảm đáng kể hàm lượng methanol trong tất cả các mẫu, với mức giảm từ 40–70% tùy theo loại rượu và thời gian xử lý. Hàm lượng polyphenol và anthocyanin dạng monomer có xu hướng giảm từ 20–80% sau khi xử lý. Hai hợp chất này thể hiện xu hướng hai pha: tăng ban đầu do phá vỡ tế bào và tăng khả năng chiết xuất, sau đó giảm do quá trình oxy hóa khi tiếp xúc lâu hoặc ở cường độ cao. Phân tích cảm quan chỉ ra rằng điều kiện siêu âm nhẹ (ví dụ 20% trong 5 phút) giúp cải thiện mùi thơm và giảm vị gắt, trong khi cường độ và thời gian cao dẫn đến thay đổi cảm quan không mong muốn. Những phát hiện này phù hợp với các nghiên cứu trước đó và cho thấy rằng việc ứng dụng siêu âm có kiểm soát có thể nâng cao quá trình lão hóa của rượu truyền thống Việt Nam, đồng thời cải thiện chất lượng và giảm hàm lượng methanol độc hại, hứa hẹn ứng dụng trong sản xuất công nghiệp.

Từ khóa: Siêu âm, lão hóa, rượu mận, rượu nếp cẩm, polyphenol, hàm lượng methanol.