Journal of Culinary Science & Technology

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/wcsc20

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To cite this article: Imran Ahmad, Muhammad B. Sadiq, A. Liu, T-A. Benjamin & Barry H. Gump (2021): Effect of Low-Frequency Ultrasonication on Red Wine Astringency, Journal of Culinary Science & Technology, DOI: 10.1080/15428052.2021.2002228

To link to this article: https://doi.org/10.1080/15428052.2021.2002228

JOURNAL OF CULINARY SCIENCE & TECHNOLOGY
https://doi.org/10.1080/15428052.2021.2002228

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Effect of Low-Frequency Ultrasonication on Red Wine Astringency

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ABSTRACT

Wine tastes better with age because of a complex chemical reaction among sugars, acids, and phenolic compounds. This study investigates if applying ultrasonic waves to wine would significantly reduce its perceived astringency levels. Ultrasonic (US) waves were applied to samples of a young Cabernet Sauvignon using a 24 kHz ultrasonic processor and *sonotrode* probe by varying time,, and amplitude at three levels. To objectively assess the relationship between the US waves and astringency, physical and chemical analysis was carried out that confirmed the underlying assumptions. While the pH of the treated samples decreased slightly, there was no change in color (Hunter CIE Color L*a*b*). Total Phenolic Content (TPC), Total Anthocyanin Content (TAC), and Tannin Concentration (TC) were significantly different ($p \leq .05$) among all samples, indicating the impact of sonication on astringency causing components. Additionally, Fourier Transform Near Infrared (FT-NIR) spectroscopy confirmed that there were notable changes in the spectra, attributed to tannins, of wine after the application of sonication in comparison to untreated wine samples. The perception of astringent flavor was evaluated by (i) expert wine tasters and (ii) untrained panelists ($n = 60$) who were able to clearly distinguish between treated and untreated samples ($p \leq .05$) and preferred sonicated samples (180 sec, 100% amplitude) over the control samples, supporting the hypothesis that sonication reduces the astringency of red wine.

ARTICLE HISTORY
Received 17 March 2021
Revised 16 September 2021
Accepted 31 October 2021

KEYWORDS
Red wine; sonication; polyphenols; anthocyanin; tannins; astringency; Ultrasound; wine aging

Introduction

As one of the most widely consumed alcoholic beverages, red wine contributed nearly 46.3% revenue to $38 billion in sales in the United States (Brager, 2016; Gordon, 2016). Market Research Future predicts a surge in the consumption of red wine due to increased demand, and the red wine market is expected to grow at a compound annual growth rate of 3.6% by 2023 (Market Research

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Future, 2019). The traditional aging method involves the storage of red wine in big barrels until the content of alcohol reaches the desired level (Carpena, Pereira, Prieto, & Simal-Gandara, 2020) and full development of flavor profile. However, the traditional method of wine aging is time-consuming and inefficient. The barrel must also be renewed over time to protect wine from the undesired growth of microorganisms such as *Brettanomyces* and *Dekkera* (Conterno, Fondazione, & Henick-Kling, 2010). Additional labor and storage costs occur in the traditional aging method, which impacts the retail products to be more expensive (Jaarsveld & Hattingh, 2016). Considering all these aspects, many researchers have focused on new methods for wine aging, which could be employed in the wine industry as an alternative to the long aging and flavor development process of wine (Saterlay & Compton, 2000).

Wine tastes better with age because of a complex chemical reaction among sugars, acids, and substances known as phenolic compounds. When a wine is young, its tannin content gives it a bitter and astringent flavor. Astringency is probably one of the most important sensory attributes of red wines. It is caused by some phenolic compounds, such as tannins, to bind salivary proteins, producing drying, and puckering sensations in the mouth (American Society for Testing and Materials [ASTM], 1989). Tannins are high molecular weight (over 500 KDa) polyphenols, precipitating with gelatin and other proteins in solution. These proteins are, in general, rich in proline (Jauregi, Olatujoye, Cabezudo, Frazier, & Gordon, 2016). The astringency of red wine is usually estimated by tasting (Valentová, Skrovánková, Panovská, & Pokorný, 2002). However, several chemical methods can be used to evaluate the astringency in red wine. These include, but are not limited to, binding proline (McRae, Falconer, & Kennedy, 2010); gelatin (Llaudy et al., 2004); ovalalbumin (Fleming, Ziegler, & Hayes, 2016); and e-tongue (Han et al., 2017).

To reduce the astringency of young red wine and improve the flavor, many researchers have come up with several physical and chemical methods, which have similar or even better effectiveness as the traditional aging. Among the chemical methods, several studies have illustrated accelerated aging by chemical methods, mostly due to the oxidation of flavonoids and pigments' polymerization (Boulton, 2001; Castellari, Matricardi, Arfelli, Galassi, & Amati, 2000). The chemical methods depend on micro-oxygenation or oxygenation (Del Álamo, Nevares, Gallego, Fernández De Simón, & Cadahía, 2010) in red wines and can stabilize color, reduce astringency and aromatic components. However, uncontrolled oxygenation can increase astringency, impart color, and promote bacterial growth. In comparison, the physical methods include storage in the oak wood barrels. Other non-conventional methods reported in the literature are the application of gamma radiation, electric field, High Hydrostatic Pressure (HHP), and ultrasonic waves (Carpena et al., 2020; Jaarsveld & Hattingh, 2016; Jackson, 2009; Sun et al., 2013; Yildirim & Dündar, 2017). Among them, HHP and Sonication treatments have received some serious consideration by

enologists and wine researchers as they were able to improve the sensorial quality of the wine, as well as improve its color after five minutes of treatment (Chang, 2005; Morabito & Leonhardt, ; Sun et al., 2015). Both the US and HHP technologies can preserve the natural ingredients and bioactive contents of fruit juices. In one study, the US has been reported to have lowered the content of Total Anthocyanin Content (TAC) in juices more than HHP (Feng et al., 2020).

The food industry has used the US technology for decades as it is a relatively low cost, non-hazardous, and environmentally friendly technology (Mason, 1996; Sun & Li, 2003). According to Soria and Villamiel (2010), ultrasonic waves within the range of 20–60 kHz promotes all the reactions that occur during the wine aging process, such as oxidation, polymerization, and condensation of alcohol, aldehydes, esters, and olefins. Several studies have also demonstrated that a US wave below 100 kHz could shorten the aging process of wine maturation (Chang & Chen, 2002; Chang, 2005; Leonhardt and Morabito, 2007).

The US's application is of interest since it provides a form of energy that is different from those normally used, such as heat, light, and pressure (Lindley & Mason, 1987). US power provides high temperature and high pressure leading to the modification of chemical reactions (Suslick, 1989), such as fragmentation and subsequent recombination of polymers (Chang, 2005). US waves, upon application to liquid samples, create acoustic cavitation of microbubbles (100 micrometers), which then collapse into localized hotspots, generating extreme heat, pressure, shockwaves, and particle acceleration in aqueous systems (Mason, 1998), similar to HHP but in a relatively smaller application area and a larger surface area. The combination of tremendous heat, pressure, and turbulence accelerates mass transfer in chemical reactions, creating new reaction pathways, breaking down particles, and generating new products from those obtained under conventional conditions (Patist & Bates, 2008). Cavitation refers to the formation, growth, and collapse of bubbles in a liquid. Suslick (1989) described the cavitational collapse as a phenomenon that produces intense local heating (~5000 K), high pressures (~1000 atm) owing to a large surface area with a small application area. The massive heating and cooling cycles with rates as high as 109 K/sec and liquid jet streams of ~400 km/h accelerate molecular disintegration and the formation of new compounds. Suslick (1989) further noted that high temperature and pressure generated through the circulating waves cause complex molecules to break down and accelerate cellular reactions. The US waves in the range of 20–100 kHz have been recommended for sonochemical reactions to take place (Lindley & Mason, 1987); 48 kHz US waves for the extraction of aromatic compounds (Cocito, Gaetano, & Delfini, 1995); and 43 kHz for fermentation control and degassing of CO_2 (Matsuura, Hirotsune, Nunokawa, Satoh, & Honda, 1994). Chang and Chen (2002) reported accelerated aging of rice and maize wines at 20 kHz and found it comparable in taste and consumer preference to conventionally aged wine.

Zhang, Shen, Fan, and García Martín (2015) recently reported that different conditions of US treatment significantly changed the total concentration of phenolic compounds and electrical conductivity. Therefore, the US is considered the most effective method for treating young, well-colored red wine that is still evolving with an immature tannic structure (Ferraretto & Celotti, 2016). As the concentrations of tannins and anthocyanins increase in wine, the aging potential of wine becomes higher.

Phenolic compounds play an important role in enology, from sensorial wine properties (flavor, color, astringency, and bitterness) to wine maturation (Spranger et al., 2004). Masuzawa, Ohdaira, and Ide (2000) concluded that the US at lower levels of applied pressure promoted the polymerization of phenolic compounds in red wine. During the aging process, three important changes occur to wine: the stabilization of wine color due to co-pigment anthocyanin complexes, the formation of new pigments, and the progressive formation of both tannin–tannin, and anthocyanin–tannin complexes (Boulton, 2001; Jackson, 2009). Similarly, Ferraretto and Celotti (2016) reported that red wines with longer US treatment durations at higher amplitudes had significant changes in their polymerization levels. They also noted that increase in temperature would have no negative consequences on anthocyanins and loss of color.

Although a significant amount of research has studied the impact of the US on phenolic compounds, the effect of the US on astringency and the sensory perception of wine has not been reported. Moreover, the treatment of wine for artificial aging purposes has a tremendous potential owing to its low cost, easy operation, and low impact as compared to higher frequency sonication, which may lead to undesirable changes to the finished wine. Therefore, the objective of this study is to investigate if applying ultrasonic waves to wine would significantly reduce its perceived astringency and improve flavor profile without impacting crucial sensorial attributes such as pH, acidity, and color.

Materials and methods

Samples

Samples of young red wine (2019 Cabernet Sauvignon) were obtained from a local distributor and stored at room temperature for further treatment and analysis.

Sonication

Ultrasonic waves were applied to red wine (Cabernet Sauvignon) samples using Hielscher UP400S (400 W, 24 kHz) with a 22 mm titanium *sonotrode* probe (Hielscher Ultrasonics GmbH). Time and amplitude were varied at three levels (exposure time: 60, 90, and 180 sec., and amplitude: 50, 75, and

100%), whereas the frequency was fixed at 24 kHz. The output temperature was recorded to ensure product is not over heated. The temperature was remained in the range of 25–30°C. The sonic wave generator was set at ON/OFF cycle at different time intervals for the duration of the treatment. To apply sonication, the *sonotrode* probe was submerged into 30 mL of red wine samples using a 50-mL beaker so that maximum exposure of sonic waves to the sample is ensured. The sonication was conducted at room temperature (25–30°C).

pH, titratable acidity, and electrical conductivity

The pH of the ten wine samples was measured using a Mettler Toledo (FiveEasyTM FE20) benchtop pH meter following Official Methods of Analysis (AOAC, 2016). The instrument was capable of measuring pH at 0.01 resolution. The meter was first calibrated with two buffer solutions of pH 4 and 7. The electrode was rinsed with deionized water between measurements. Similarly, the Oxidation-Reduction Potential (ORP), mV, was determined separately by the same pH meter. The pH and ORP of 100 mL of treated and untreated wine samples were measured in triplicate. All measurements were done at room temperature (25°C).

Titratable acidity (TA) was determined for predominant acids found in red wine, tartaric acid, and malic acid using 0.1M NaOH solution (Zoecklein, Fugelsang, Gump, & Nury, 2013). Briefly, 200 mL of distilled water was placed in a 500-mL Erlenmeyer flask, and 1 mL of phenolphthalein indicator was added. Distilled water was titrated against 0.1M NaOH until the endpoint marked by pink color was observed. The 5 mL of wine sample was added to the flask. Finally, the sample was titrated with 0.1M NaOH until the same pink end point was observed. The volume of NaOH used up during titration was noted, and TA was determined as tartaric acid using the following formula (Eq. 1):

$$TAastartaricandmalicacid\left(\frac{g}{100}ml\right) = \frac{V_{NaOH} \times M_{NaOH} \times 75 \times 100}{1000 \times V_{sample}} \quad (1)$$

Where V represents the volume of NaOH and sample while M represents the molarity of NaOH, determined previously, all measurements were conducted in triplicate, and results are reported in terms of g/L.

Color measurement

Total Color Difference (TCD)

CIE Color Lab values (L-value: lightness, a-value: redness and greenness, b-value: yellowness and blueness, chroma, hue angle), using a Hunter Color meter. The instrument was standardized according to the CIE (Commission

International de l' Eclairage) using a standard white reference tile (calibrated as L* 93.33, a* −0.91, b* 1.46). The average L, a, and b values are then converted into total change in color (ΔE), according to the following formula (Eq. 2):

$$\Delta E = \sqrt{\left[(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2\right]} \quad (2)$$

where L_1, a_1, and b_1 are the initial color values of control samples. The corresponding L_2, a_2 and b_2 show the color values for a given treatment.

Color intensity

Wine color intensity was measured in terms of absorbance of incident light using a UV-Vis spectrophotometer (Molecular Devices Inc.) within the visible spectrum (420–620 nm) according to the Beer-Lambert law (Atkins & de Paula, 2006). The color intensity is given by the sum of absorbance measurements in the violet, green, and red areas of the visible spectrum (Eq. 3):

$$\text{Wine color intensity} = A_{420} + A_{520} + A_{620} \quad (3)$$

where A_λ denotes the absorbance at wavelength λ.

Prior to measurements, the spectrophotometer was zeroed using deionized (DI) water as blank. Undiluted wine samples were subjected to absorbance measurements by using a 0.1-cm thickness cell. The absorbance was then multiplied by 10, as described by Zoecklein et al. (2013). An additional parameter, hue, was determined using the ratio of absorbance at 420 and 520 nm wavelengths.

Astringency measurement

Tannin content

The concentration of proanthocyanidins in the aqueous extract was determined by Hagerman's acid butanol assay for proanthocyanidins (2002). Briefly, 0.2 mL of the iron reagent (2% ferric ammonium sulfate in 2M HCl) and a 1 mL sample of extract were added to 6 mL of the Acid Butanol reagent (950 mL of n-butanol and 50 mL of concentrated HCl). The solution was vortexed and then incubated for 50 min in boiling water. Absorbance was read at 550 nm while using a Microplate Reader (SpectraMax 190 Molecular Devices, Sunnyvale CA, USA). A representative standard curve of proanthocyanidins concentration versus absorbance was used as a reference (Sintara et al., 2018).

Phenolic content

The sample preparation for polyphenol extracts was achieved using Solari-Godiño's (2017) procedure, with some modifications. 1 mL of sonicated wine samples were mixed with 9 mL of methanol in a glass beaker and stirred with a magnetic stir bar at 400 RPM for 30 minutes at room temperature, with minimal light exposure. The solution was then filtered through a non-sterile 45-µm MCE membrane syringe filter. The supernatants were then stored at 4°C until further use. Control samples were prepared similarly but were not filtered.

Total phenolic content (TPC) of each sample was determined using the Folin-Ciocalteu (F-C) reagent according to the method of Waterhouse (2003), with some modifications. The red wine sample was diluted with distilled water in a ratio of 1:10 (v/v). Briefly, 780 µL distilled water was mixed with 50 µL of F-C reagent and 20 µL of the diluted sample. After 1 min of resting time, 150 µL of Na_2CO_3 (20% w/v) was added. The flask was swirled to mix and incubated at room temperature for two hours. 200 µL were transferred into the wells of a 96-well microplate reader (SpectraMax 190, Absorbance Microplate Reader Molecular Devices, Sunnyvale CA, USA), and the absorbance was at 765 nm was recorded by repeating at least 9 readings (replicates). Readings were multiplied by a dilution factor of 10 for the correct concentration. Gallic acid standard curve for calibration and control samples also followed the above protocol. Values of Gallic acid equivalents (GAE) are reported using mg/L for wine samples.

Anthocyanin content

For the determination of the total anthocyanin content (TAC), Somers and Evans's method was used (Nieuwpoort and Buica, 2017) with some modifications incorporating Australian Wine Research Institute [AWRI's] (2012) High-Through-put (HTP) assay. In a test tube, 0.02 mL of red wine sample was combined with 1.08 mL of 1M HCl. In a separate test tube, 0.02 mL of wine with 1.08 mL of aqueous SO_2 compound was mixed. Both tubes were incubated in the dark for 3 hours. Absorbance was read at 520 nm in a 96 deep-well Microplate Reader (SpectraMax 190, Molecular Devices, Sunnyvale CA, USA), with at least 8 replicates. Plates were sealed and shaken gently, on automated plate shaker mode, to allow mixing prior to incubation.

Calculations (Nile, Hwan Kim, & Keum, 2015):

$$TAC\left(\frac{mg}{l}\right) = 20 \times [(50 \times A_{520}HCl) - (1.6667 \times (10 \times A_{520}SO_2))] \quad (4)$$

Fourier transform near infra-red spectroscopy (FT-NIR)

The wine samples, untreated and sonicated, were subjected to FT- NIR analysis using Nicolet^{TM} iS FT-NIR Spectrometer (ThermoFisher Scientific) to observe the spectral changes in near infrared region (12500–4000 cm^{-1})

(Camps, Steffen, Quennoz, Simonnet, & Gilli, 2017; Power et al., 2021; Noypitak, et al., 2015.) Absorbance spectra (average of 32 scans) were captured at a resolution of 8 nm. The spectral data were analyzed using OMNIC Software (ThermoFisher Scientific).

Sensory analysis

The sensory analysis was conducted using an expert panel and untrained panelists.

Three experienced enologists were requested to objectively assess control and sonicated samples for texture (roughness vs. smoothness), moistness vs. dryness, and puckering sensation on a 5-point scale, in addition to open-ended feedback.

For the consumer preference test, sixty (60) untrained panelists of all ages and gender were assigned a unique randomly generated tray code with coded samples. On the day of the experiment, the samples were sonicated, according to the scheme outlined above, an hour before the investigation. To properly regulate the temperature of the samples, all of them were stored in three identical wine bottles at 55 F (12.7°C) for 45 minutes. They were then poured right into cups before serving.

Duo-trio test

The Duo-trio test was conducted in 2 triads. In the first round, 60 panelists were presented with three samples – two controls and one sonicated – to taste in order from left to right. The first sample – the reference – was always the control. The last two were randomly coded for each panelist. In addition to the three samples, they were also provided with a small cup of water and half a slice of bread to cleanse their palates in between. All three samples were served at the same temperature of 60F (15.5°C). Panelists were asked to match one of the coded samples to the reference. The same procedure was repeated with the second triad of 60 panelists.

Paired Preference Test (2AFC)

Sixty panelists were provided with two samples: control and sonicated. After assessing the first sample, they were clearly instructed to use bread and water to cleanse their palates before proceeding to the next sample. Then they were asked to answer which sample they preferred. All samples were served at 60°F (15.5°C).

Choose-only-one test

Panelists were asked to compare control and sonicated samples for texture (rougher vs. smoother), moistness (dry sensation), and puckering sensation. The data were reported in percent of panelists preferring one sample over the other for the above-given attributes.

The sensory analysis was set up and administered using Compusense® (Compusense Inc. Guelph ON, Canada). Collected data were then analyzed using D-prime analysis (for the duo-trio test, $\alpha \leq 0.05$) and paired t-test for paired preference test ($\alpha \leq 0.05$). The experiment was conducted in the Wine Tasting laboratory at Florida International University.

Data Analysis

Results were expressed by means ± standard deviation of six or more separate determinations. Comparisons of means were performed by one-way Analysis of Variance (ANOVA), and a t-test of independence was used to determine the differences in the mean value ($p \leq .05$). Data were analyzed using XL-STAT® software.

Results and discussion

Titratable acidity, pH, and electrical conductivity

Titratable acidity is a representation of predominant organic acids present in the must. Tartaric, malic, and citric acid account for 90% of acidity and largely depends on the grape variety and fermentation process. The acid-base titration method is a good indicator of organic acids. The TA was found to be in the narrow range of 0.6–0.7 g/L, but there was no clear and significant difference found among control and sonicated samples at any level. On the other hand, both the pH and ORP exhibited varying levels of significance ($p \leq .05$) as both the time and amplitude levels increase (Table 1). The pH of the control sample was 3.71, which is normal for low-acid red wines, but the sonicated samples pH decreased to 3.62 as both the time and amplitude levels increased. However, this small change without any clear trend cannot be attributed to sonication, as evident by TA. There is no direct relationship between pH and TA, but higher acid levels may generally be associated with lower pH. Since the samples were drawn from finished 'commercial' wines, any difference between TA and pH can be ruled out. Chang and Chen (2002) reported mixed results when they applied 20 kHz US waves on rice and maize wines. While there was a slight increase in the pH of rice wine, the pH of maize wine remained the same. Therefore, it can be concluded that US waves in the range of 20–24 kHz may not change the pH of wines and, consequently, will not affect the sourness of wines – a much desirable trait by enologists.

Table 1. Titratable acidity, pH, and electrical conductivity of control and sonicated red wine samples[††].

Treatment[†]	Titratable Acidity (g/L)	pH	Oxidation-Reduction Potential (mV)
Control	0.70[a]	3.71[a]	196.0[b]
US60-50	0.60[a]	3.64[ab]	199.7[ab]
US60-75	0.65[a]	3.68[ab]	198.0[ab]
US60-100	0.60[a]	3.66[ab]	198.0[ab]
US90-50	0.60[a]	3.66[ab]	199.0[ab]
US90-75	0.63[a]	3.63[b]	200.7[a]
US90-100	0.60[a]	3.62[b]	201.7[a]
US180-50	0.65[a]	3.64[ab]	200.3[ab]
US180-75	0.66[a]	3.63[b]	201.0[a]
US180-100	0.60[a]	3.62[b]	201.0[a]

[†]Ultrasonic application time (sec)-amplitude (%).
[††]TA (Sig. $p \geq 0.05$), pH, and EC (Sig. $p \leq .05$) (Different superscript letters indicate the difference in the level of statistical significance).

Obreque-Slier, Peña-Neira, and López-Solís (2012) reported that wines at pH 3.5 were perceived as more astringent via a trained sensory panel. However, expert panelists (Table 2) described the control sample as having sharper (more bitter) tannins than the sonicated samples, which were described as having a smoother, rounder (less astringent) finish. Similar studies reported that ultrasound frequency and power had no significant effect on pH and ORP (Zhang et al., 2015). However, it is noted that the higher amplitudes result in more effective creation of cavitation, as evident by the data (Table 1).

Effect of sonication on color of red wine

As shown in Table 2, no significant changes in L* (lightness), a* (red/green coordinate), b* (yellow/blue coordinate) values were observed in wines treated by sonication ($p \geq 0.05$). Likewise, Color Intensity (CI) and Hue did not

Table 2. The total color difference, color intensity, and hue of wine samples[††].

Treatment[†]	L*	a*	b*	ΔE[§]	C.I (A_{λ}) [§§]	Hue[§§§]
Control	32	16	8	0.00	5.73[a]	0.82[a]
US60-50	34	20	11	4.376	5.94[a]	0.8[a]
US60-75	34	21	12		5.75[a]	0.81[a]
US60-100	32	22	11		5.81[a]	0.8 [a]
US90-50	35	22	11	3.72	5.81[a]	0.82[a]
US90-75	32	21	10		5.78 [a]	0.82[a]
US90-100	35	23	11		5.82[a]	0.82[a]
US180-50	34	22	11	3.21	5.75[a]	0.82[a]
US180-75	34	21	9		5.77[a]	0.82[a]
US180-100	34	23	10		5.74[a]	0.83[a]

Lab* – CIE Lab Color attributes
[†]Ultrasonic application time (sec)-amplitude (%)
[††]The letters in superscript indicate a lack of statistical significance at $p > 0.05$
[§] ΔE – Total Color Difference
[§§]CI – Color Intensity
[§§§]Hue is dimensionless

change significantly and were close to the control samples, confirming that US treatment does not adversely impact the color of the wines. The Total Color Difference (TCD, ΔE) based on Euclidean distance from the control samples, in terms of CIELab parameters, were 4.37, 3.72, and 3.21 for 60, 90, and 180 sec of US exposure, respectively. This slight difference is usually unperceivable by consumers and might be due to the high measuring resolution of the instrument (Mokrzycki and Tolol, 2011). A decreasing trend in coordinate a* is a process seen in the natural aging of wines; however, a majority of the samples sonicated for shorter periods of time at lower amplitudes demonstrated an increasing trend. The increase of a* indicates the role sonication plays in the enhancement of redness in wine and the loss of color intensity that occurs during aging (García Martín & Sun, 2013; Zhang, et al., 2016). Celotti et al. (2020) also noted that a decrease in color intensity is indicative of long sonication treatments, which can negatively affect the wine aging process. However, it is safe to say that 24 kHz sonication did not adversely impact the wines as compared to the natural aging process.

Phenolic content

Total polyphenols present in control and sonicated samples are shown in Table 3. Results ranged from 0.154 to 0.230 mg/L for control to 180 sec of sonication of wine samples and were significantly affected by exposure time ($p < .05$), indicating that the US induced extraction of phenolic compounds. Waterhouse (2003) attributes production techniques and high temperatures to the increase of phenolic compounds during extraction. Red wines should exhibit TPCs of 1 to 3 g/L, with the typical average of ~1.8 g/liter, yet wine samples were significantly lower than Waterhouse's reported TPCs. However, our findings were in accordance with the research reported by Ferraretto and Celotti (2016), who mentioned how Ultrasound could affect the polymerization of polyphenolic compounds in red wine at low amplitudes. The results of F-C assay show that sonication influences TPCs in red wine. Samples that were

Table 3. Total polyphenol content, total anthocyanin content and tannin concentration of red wine samples[††].

Treatment[†]	TPC (mg/L)	TAC (mg/L)	TC (mg/mL)
Control	0.207^{b}	130.73^{a}	1.810^{a}
US60-50	0.194 ^{c}	136.58^{a}	1.749^{a}
US60-75	0.166^{d}	137.25^{ab}	1.725^{a}
US60-100	0.163^{de}	108.52^{ab}	1.620^{b}
US90-50	0.211^{b}	149.72^{ab}	1.602^{b}
US90-75	0.211^{b}	135.66^{ab}	1.369 ^{c}
US90-100	0.230^{a}	126.30^{ab}	1.295 ^{cd}
US180-50	0.193 ^{c}	143.38^{ab}	1.147^{de}
US180-75	0.208^{b}	139.66^{ab}	0.994^{ef}
US180-100	0.154^{e}	139.10^{b}	0.935 ^{f}

[†]Ultrasonic application time (sec)-amplitude (%)
[††]Sig. p ≤ .05 (the different superscript letters indicate statistical significance).

sonicated for 90 seconds had higher levels of TPCs than the control sample as amplitude level increased (Table 3). However, samples that were sonicated for 60 seconds exhibited a decrease in TPCs as sonication amplitude levels increased. Samples that were sonicated for 180 seconds exhibited a similar trend to samples sonicated for 60 seconds, except for the sample sonicated for 180 sec at 75% amplitude. Therefore, the impact of variable amplitude on TPCs was inconclusive, as evident by the data (Table 3).

Anthocyanin content

Nieuwoudt's modified Somers assay (2001) for TAC, obtained from the sonication method used for young Cabernet Sauvignon, showed some significant differences ($p < .05$) from the control. The TACs in control samples were lower than the AWRI (HTP) assay (2012) results, but sonicated samples at 50% amplitude for 90 and 180 seconds were significantly greater. Most of the wine samples have greater anthocyanin content (mg/L) than the control, aside from US 60–100 and US 90–100 (Table 3). Unlike Ferraretto and Celotti's (2016) sonicated Cabernet Sauvignon samples, our sonicated samples produced significantly lower anthocyanin content (mg/L). This is possibly due to the samples being sonicated at higher amplitude and at relatively shorter periods of time (Table 3).

Anthocyanins are one of the main classes of flavonoids and are responsible for the color of red wines. The pH of wine can affect the initial color of wine and progressively change during the wine aging process (Soares, Brandão, Mateus, & De Freitas, 2017). Sonicated samples did not differ significantly in color from each other or from the control sample, as shown in Table 2. Aside from the total color difference, the color intensity and hue did not show any significance ($p > .05$) in color between treated samples and the control. While sonication does not exhibit drastic effects on color, the change in pH values indicates that at low amplitudes, Ultrasound can have some effect on the polymerization of polyphenolic compounds in red wine Ferraretto and Celotti (2016).

Tannin content

Tannin content is considered to have a direct relation with puckering sensation attributed by consumers as astringent feeling in the mouth. The results revealed that sonication at 24 kHz significantly lowered tannin content, as determined by proanthocyanidins assay. As the amplitude of sonic waves increased, the total tannin content in the sonicated wine samples decreased. The 180 sec treatment time yielded the lowest tannin content as compared to samples sonicated for 60 and 90 seconds. The amplitude levels of less than 100% seem to have no impact on tannin content (Table 3). As tannins are

formed by the polymerization of flavanols, in order for tannins to form stable complexes with protein, their molecular weight should be in the range of 600–3500 kDa. The acoustic cavitation phenomenon has a possible contribution to depolymerization (Liu et al., 2020), resulting in lower astringency levels. Intermolecular oxidation reactions, such as cavitation and bubble formation, yield many lower molecular weight compounds (between 2 and 5 KDa) (Khanbabaee and van Ree, 2001), which eventually are unable to bind with saliva proteins. The phenomenon also applies to the condensed tannins formed during aging in barrels. However, hydrolyzable tannins form other acids such as Ellagic acid – one of the most important indicators for the wine aged in barrels.

FT-NIR spectrometry

The taste and nutritional value of wine is greatly influenced by the phenolic compounds. Most of the phenolic compounds such as anthocyanins do not interfere with mouthfeel of wine. However, tannins and flavan-3-ols are associated with bitter and astringency of wines (Buratti et al., 2011). The Figure 1 illustrates that there were notable changes in the spectra of wine after the application of sonication (exposure time: 60, 90, and 180 sec., and amplitude: 50, 75, and 100%) in comparison to untreated sample. With the increase in sonication time there were more profound changes in spectral data, indicating that sonication resulted in the modification of chemical constituents of wine. The spectral region 6500–5700 cm^{-1} corresponded to phenolic compounds and the absorption band 7330–7200 cm^{-1} were attributed to tannins (Aleixandre-Tudo, Nieuwoudt, Aleixandre, & du Toit, 2018). Similarly, Dykes, Hoffmann, Portillo-Rodriguez, Rooney, and Rooney (2014) reported the absorption bands at 1415–1512 nm (7067–6613 cm^{-1}), 1650–1750 nm (6060–5714 cm^{-1}), and 1955–2035 nm (5115–4914 cm^{-1}) corresponding to polyphenols, flavonoids, and condensed tannins. The wine samples subjected to sonication showed a marked decrease in absorption band (7330–7200 cm^{-1}), corresponding to the tannins. The increase in sonication time resulted in a decrease in tannin absorption band of wine samples. Noypitak, et al., 2015reported consistently lower average absorption spectra for de-astringent permission than astringent samples.

Sensory analysis

Expert panel

The overall results from the expert panels showed that the sonicated sample was perceived as being less puckering, attributing it to having a balanced finish and round tannins (Table 4 and 5). Thus, confirming the impact of US waves

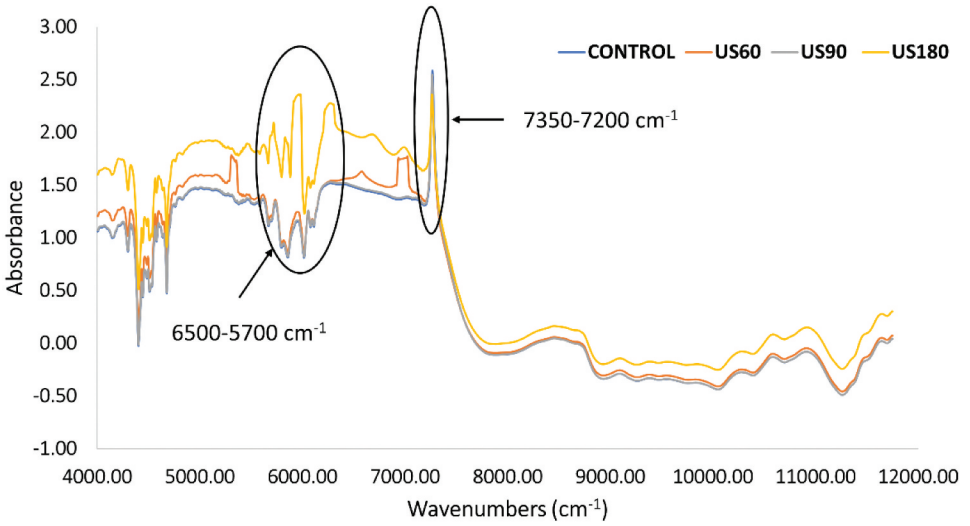

Figure 1. FT-NIR spectra of treated and untreated red wine samples (12500–4000 cm^{-1}). (sonication exposure time: 60, 90, and 180 sec., and amplitude: 50, 75, and 100%).

on the perception of astringency sensation in red wines. The scores for texture, moistness and sensation of control sample were 2.40 ± 1.14, 2.80 ± 0.84 and 2.60 ± 0.55, respectively, whereas the same parameters were observed in sonicated wine samples with scores of 3.40 ± 1.47, 3.20 ± 0.84 and 2.80 ± 0.45, respectively. All 5 panelists agreed on the fact that control and treated samples had a different flavor profile, as evident by their comments. However, the Likert scale was not conclusive. Mean scores for texture, moistness and sensation (puckering) were consistently higher for sonicated samples than the control samples (Table 4). The mean scores for 'texture' were 2.40 and 3.40 for control and sonicated, respectively, and had the highest standard deviation deviation (47.5%) for both control and sonicated samples indicating variation in assessing mouthfeel (Table 5). Whereas, wine samples were clearly distinguishable in terms of 'moistness' and 'sensation' mean scores with lower standard deviation than for 'texture. Briefly, the responses of expert panel to open ended questiossn were; 3/5 panelists found the control sample as rougher than smooth, while the other 2 panelists did not find it different. For moistness, one panelist was undecided, one found control as more moist than dry, while the other three panelist found the sonicated sample to be moister.

Table 4. Mean scores of sensory evaluation of control and sonicated red wine samples by an expert panel (n = 5) [††].

Attribute	Control	Sonicated
Texture	2.40 ± 1.14	3.40 ± 1.47
Moistness	2.80 ± 0.84	3.20 ± 0.84
Sensation	2.60 ± 0.55	3.80 ± 0.45

Table 5. Sensory evaluation of control and sonicated red wine samples by an expert panel [††].

	Expert Panelist A		Expert Panelist B		Expert Panelist C		Expert Panelist D		Expert Panelist E	
	Control	Sonicated[†]	Control	Sonicated[†]	Control	Sonicated[†]	Control	Sonicated[†]	Control	Sonicated[†]
Comments	Good acidity, clean finish	Balanced, clean finish	Longer finish, less aroma	Felt big, raspberry aroma	Dryer, sharper tannins	Smoother, rounder tannins	Rough than sonicated	Smoother than control	Dry puckering	Balanced

[†]Sonicated refers to US (24 kHz, 180 sec, 100% amplitude)
[††]Wine samples were rated on a 5-point scale based on texture (1 = very rough, 5 = very smooth), moistness (1 = very dry, 5 = very moist), and sensation (1 = most puckering, 5 = least puckering)

Likewise, for the puckering sensation, one was undecided; three perceived the sonicated sample as less puckering, and the other preferred the control sample (as less puckering). The sonicated sample was perceived as having less astringency with a better mouth-feel. The expert panelists attributed the smoothness of the sonicated sample to bigger and rounder tannins. This finding is consistent with the fact that tannins form long chains with each other or polymerize with monomeric anthocyanins as wine ages, thereby becoming larger and heavier, making the wine less astringent with a smoother mouth-feel. The smoother mouth-feel resulted from big tannins having a less reactive surface to readily bind to proteins in the saliva. The expert panel findings support the existing research that sonicated red wines have significant changes in their polymerization levels and astringency (Ferraretto & Celotti, 2016). As for the moistness of samples, the sonicated sample felt a bit more drying than the control in the mouth. According to the responses from the open-ended question regarding the mouth-feel of each sample, the sonicated sample was described as a balanced wine with bigger, smoother, and rounder tannins, whereas the control was evaluated as having a dryer and sharper tannins with good acidity and *longer* finish.

The duo-trio test

The chance of selecting the correct response was one out of two. Table 6 summarizes the results of both triads. In both triads, the majority of panelists (55% and 60%) were able to correctly identify the treated sample by matching the coded samples to the reference. Based on the D-prime table, C – the measure of bias was zero. The d′ values – the measure of discriminability, were 0.25 and 0.507, respectively, for triad 1 and 2. Based on d′, it is concluded that the duo-trio test was neutral.

Preference test

For the paired preference test, 73% of panelists indicated that they preferred the treated wine samples over the control sample. A significant difference between the two coded samples in terms of preference [$d' = 0.88$, $p < .05$] was noted (Table 7).

Table 6. D-prime table of constant-reference Duo-trio test.

Sample 1 – Sample 2	Chance	N	Correct	Incorrect	d′	C	Decision
Triad 1	1 in 2	60	33	27	0.252	0.00	neutral
Triad 2	1 in 2	60	36	24	0.507	0.00	neutral

d′ – the measure of discriminability.
C – the measure of bias.

Table 7. D-prime table of the paired preference test.

Attribute	Sample 1 chosen	Sample 2 chosen	d′	Avg Diff (2–1)	p-value	Sig.
Paired Preference	44	16	0.88	−0.47	0	Yes

To further elaborate the reasoning of panelists as to why the control and treated samples were different, a chose-only-one series of questions was presented to panelists. The comparison of attributes for the preferred samples was: smoother vs. rougher; slippery (moistness) vs. drying sensation; and less puckering vs. more puckering sensation (Figure 2). Of the 44 panelists who preferred the sonicated sample over the control sample, 63% assessed it as smoother than rougher, 61% described it as more slippery in the mouth with less of a drying sensation, and 66% thought of it as less puckering than the control sample. On the other hand, the control sample consistently received lower approvals for the desired attributes. Therefore, the results from all three sensory tests supported the hypothesis that sonication would reduce the perceived astringency of the wine.

Conclusion

The repeated sensorial analyses, expert panel; duo-trio and; preference test; and choose-only-one tests confirmed that the sonicated sample was perceived as having less astringency with a better mouth-feel. Both the expert and

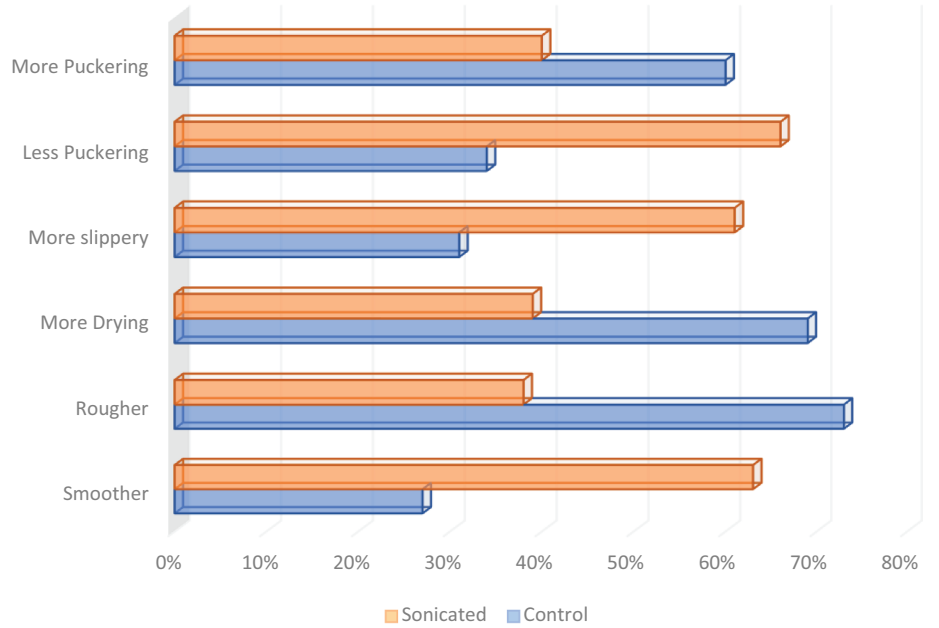

Figure 2. Sensory preference test (2AFC) for control and 24 kHz sonicated (180 sec., 100% amplitude) samples.

untrained panelists attributed the smoothness of the sonicated sample to bigger and rounder tannins. This attribute is in line with tannins' ability to form long chains with each other and polymerize with monomeric anthocyanins as wine ages. It was thereby tannins becoming more extensive and more complex, making the wine less astringent with a smoother mouth-feel. The smoother mouth-feel resulted from complex tannins having a less reactive surface to readily bind to proteins in the saliva. The results of the sensory panel support the existing research that sonicated red wines have significant changes in their polymerization levels and astringency (Ferraretto & Celotti, 2016). The qualitative analysis of astringency causing compounds using FT-NIR spectra supported the hypothesis of the study as with the increase in sonication time there were more notable changes in spectral data, suggesting that sonication resulted in the modification of phenolic compounds and tannins.

Moreover, the US waves in the range of 24 kHz had minimal effects on the color intensity and hue, and acidity profile of samples. As there were minimal changes in TAC, leading to panelists having less astringency with a better mouth-feel without affecting the color of the wine. Another benefit of using a low ranging frequency of ultrasonic waves is the extraction of aroma compounds. Vila, Heredia Mira, Beltran Lucena, and Fernández Recamales (1999) reported simultaneous extraction of several aromatic compounds using the ultrasonic method for the purpose of varietal differentiation.

Finally, the findings of this study indicate enough evidence of the effects of the US application on red wine. The exposure time of 180 sec with 100% amplitude has an adequate and positive impact on the flavor profile. However, the lower exposure times (60–90 sec) and amplitude (50 to 75%) did not significantly impact any attributes. Future research should explore the possibility of a longer duration of US exposure. Longer exposure duration could potentially have a bigger impact on tannin polymerization levels. However, caution should be given on extreme temperatures during cavitation, which may negatively impact the body, flavor, and aroma profile. Moreover, measuring astringency using sensory analysis techniques offers considerable challenges as astringency is not a true taste dimension but rather a tactile sensation. As a word of caution, the panelists should be clearly instructed that they should not assess samples in quick succession without adequate palate cleansing. Astringency is one of the in-mouth sensations that develops very slowly, approximately 15 seconds, to reach maximal intensity. It takes even longer for perceived intensity in the mouth to decline – the intensity and duration of astringency increase with repeat sampling. Without adequate palate-cleansing, the panelists could make a sequence of similar sensations.

The application of low-frequency US waves is a low-cost and easy to implement technology in an in-line setup where the exposure time is adjusted by controlling the flow of wine in pipes. This could bring about the same effects on sensorial wine properties as the natural aging process and would be

a breakthrough for the wine industry. Considering the US is a green and clean technology that will have a huge impact on industrial operations by saving labor costs, space, and energy.

Disclosure statement

No potential conflict of interest was reported by the authors. The use of human subjects was approved by the Institutional Review Board [IRB-18-203].

Funding

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