Vitis **58** (Special Issue), 83–88 (2019) DOI: 10.5073/vitis.2019.58.special-issue.83-88

Ultrasonic treatments during the alcoholic fermentation of red wines: effects on 'Syrah' wines

A. Ruiz-Rodríguez, C. Carrera, M. Palma Lovillo and C. García Barroso

Agrifood Campus of International Excellence (CeiA3), IVAGRO, Department of Analytical Chemistry, Faculty of Sciences, University of Cádiz, Puerto Real, Cádiz, Spain

Summary

Grapes grown in warm climates have shorter ripening periods. This makes the regular level of several compounds related to sensory properties in wine to remain low. Therefore, those grapes need to receive a particular treatment during winemaking processes if they have to reach the adequate content levels of colour and aroma related compounds. Applying ultrasound during some of the winemaking procedures may contribute to improving the maceration process, which would result in a higher recovery of compounds from grape skins and seeds to the must. This work studies the effect of applying ultrasound to 'Syrah' musts for two different time lengths during its alcoholic fermentation. The wines produced according to regular winemaking procedures (reference wines) were then compared to wines that had been produced under the effect of ultrasound for 30 to 60 min per day. The results showed that the wines produced using ultrasound had concentrations of volatile compounds higher than their reference wine. These data were consistent with the results from the tasting panel, where the judges highlighted the red fruit notes of the wines resulting from the application of ultrasound during the alcoholic fermentation. On the other hand, there were differences between the two wines resulting from applying ultrasound for two different lengths of time, applying ultrasound for 30 min per day proved to be more effective in terms of aroma than applying ultrasounds for 60 min per day.

The conclusion of this research is that applying ultrasound during the alcoholic fermentation favours the extraction of volatile compounds. However different times can produce different results. Furthermore, an excessive application of ultrasound may lead to the degradation of some of the compounds of interest.

K e y w o r d s : ultrasounds; volatile compounds; 'Syrah'; red wine; grape.

Introduction

In the traditional production of red wine, the alcoholic fermentation phase is directly related to the incorporation of some of the components in the solid parts of grapes into the grape juice in order to provide wine with its characteristic properties. The extraction results depend on several factors, such as ethanol concentration, cap management and temperature. These factors can be managed to provide optimum conditions for the alcoholic fermentation (Sacchi *et al.* 2005, Bosso *et al.* 2009, González-Neves *et al.* 2012). Wineries often use other procedures, before the alcoholic fermentation, as a simple way to selectively extract aromatic components and anthocyanins that may have a significant benefit on the resulting wine (Álvarez *et al.* 2009). For example, pre-fermentative maceration at low temperature, produced wines with more colour in some cases (Casassa *et al*. 2015). Another study on the effect of prefermentative maceration on 'Monastrell' and 'Cabernet Sauvignon' grape varieties showed increased levels of proanthocyanidins in the final wines (Busse-Valverde *et al.* 2010). Regarding 'Syrah' wines, different pre-fermentative maceration times directly affect their phenolic content. (Cejudo-Bastante *et al.* 2014). The effects of pre-fermentative maceration was evaluated on 'Cabernet Sauvignon' grapes for a period of 10 d at 10 °C before starting the alcoholic fermentation. Using these conditions, the contents of anthocyanins, catechin and epicatechin clearly increased in comparison to the traditional process (Panprivech *et al.* 2015).

New options have been proposed over the last few years to enhance the effects of pre-fermentative maceration. Ultrasound has already been proposed as an interesting option to speed up extraction processes. At laboratory level, works have been developed that focus on how to extract aromatic components by means of ultrasounds (Vila *et al.* 1999, Hernanz *et al.* 2008). The effect of ultrasound on other compounds of interest have also been studied. A study on the use of ultrasound with 'Syrah' grapes proved to be an alternative for the extraction of total phenolic compounds and anthocyanins (Mazza *et al.* 2018).

Ultrasound can be applied by means of different type of equipment and for different lengths of time (El Darra *et al.* 2013). In this study, the different effects of applying ultrasound for different time lengths were tested in order to select the optimal treatment conditions with a reduced energy consumption. Short ultrasound treatments (15 min) were more effective than moderate treatment in terms of the extraction of phenolic.

This beneficial effect of ultrasound on some of the winemaking processes has been successfully extended to wine

Correspondence to: Dr. A. Ruiz-Rodríguez, Agrifood Campus of International Excellence (CeiA3), IVAGRO, Department of Analytical Chemistry, Faculty of Sciences, University of Cádiz, P.O. Box 40, 11510 Puerto Real, Cádiz, E-mail: ana.ruiz@uca.es

aging. Really interesting results regarding the extraction of phenolic compounds from woods into red wines and spirits have been obtained (Chang 2005, Ferraretto and Celotti 2016, Zhang and Wang 2017). Optimum conditions for the extraction of phenolic compounds from solid matter seems to be at 10-15 % ethanol in the liquid media and ultrasounds (Zheng *et al.* 2014, Zhang and Wang 2017).

The present study intends to evaluate the effect of applying ultrasound for different lengths of time to 'Syrah' grapes must during its fermentation phase with regards to the extraction of phenolic compounds, anthocyanins, tannins and aroma compounds.

Material and Methods

Wine production: Approximately 750 kg of grapes from *Vitis vinifera* L. 'Syrah' were manually harvested at their optimum maturity phase (22.84 °Brix) from vineyards in the province of Cadiz (Spain). The grapes were stored at 4 °C for 24 h before processing.

The grapes were weighed (average weight = 1.54 g per berry), destemmed, and crushed. 50 L tanks were used for the fermentation process. Experiences were run in duplicate. To prevent microbiological activation, sulfur dioxide was added to the pre-treated must (30 $mg \cdot L^{-1}$). Tartaric acid was used to adjust the pH at 3.6. Then, *Saccharomyces cerevisiae* yeast (Viniferm Elegancia, AGROVIN, Alcázar de San Juan, Spain) was added (20 $g \cdot hL^{-1}$) to the musts. The alcoholic fermentation began within 24 h of crushing. Fermentation temperature was fixed at 24 °C. Fermentation tanks were allocated in a special fermentation chamber with specific temperature control. Temperatures at each fermentation tank were checked twice a day, values between 22.8 and 24.3 °C were recorded during the fermentation period.

The reference wine (REFERENCE) was obtained according to traditional winemaking procedures, whilst US-30MIN and US60MIN wines were obtained by applying ultrasounds for 15 or 30 min respectively twice a day until the pomace was pressed, *i.e.* for 16 d. The cap was punched down twice a day throughout the whole process, just before applying ultrasounds to the US30MIN and US60MIN tanks (Fig. 1).

The ultrasound was applied by immersing the tanks in a water bath inside an ACM-200E system, a 28 KHz - 2 KW system by ULTRATECNO (Valencia, Spain). The temperature was set up at constant 24 °C. Temperature in the fermentation tank was checked at the end of the application of ultrasound, it reached values not higher than 24.3 °C.

After the alcoholic fermentation (10 d) the grape pomace was in contact with the wine for 6 d, then it was lightly pressed (1 $kg \cdot cm^{-2}$). The wines were then allowed to further settle and cold-stabilize at 4 °C for 4 weeks prior to bottling, and 80 $mg \cdot L^{-1}$ of sulphur dioxide was added to obtain a free sulphur dioxide concentration of 30 to 40 $mg \cdot L^{-1}$. The 6 wines were bottled separately into 750 mL Bordeaux-style dark amber glass bottles. All the wines were stored at 20 °C until their analysis or tasting trials.

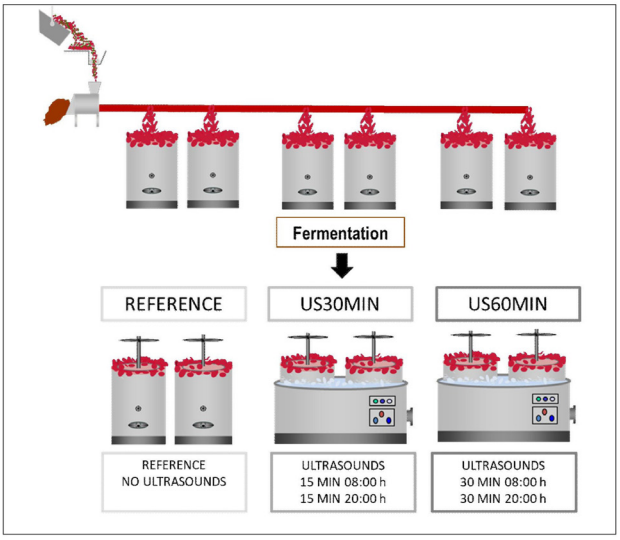


Fig. 1: Winemaking conditions.

Analytical control of the musts and wines: Both the musts and the wines have been characterized by determining regular parameters (sugar content, ethanol, pH and total acidity) as well as polyphenols, anthocyanins and total tannin content. The density measurements were carried out in a densimeter DMA 4500M (Anton Paar, Graz, Austria). Acidity was calculated by acid-base titration using a pHmatic 23 automatic tritrator (Crison, Düsseldorf, Germany). The red colour was determined directly by absorbance measurements at 520 nm after a month of alcoholic fermentation and also during storage in the bottles.

Anthocyanins and total phenolic compounds and tannin contents were measured following the protocol for the determination of phenolic maturity based on the method developed by the Australian Wine Research Institute (AWRI) Gishen *et al.* 2005, Carrera *et al.* 2012) with minor modifications. To measure anthocyanins and total phenolic compounds in the sample, a solution of 1M HCl was added and the absorbance was recorded at 520 and 280 nm respectively by means of a UV/VIS spectrophotometer V-530 (Jasco, Madrid, Spain).

For the determination of total tannins, the sample was treated with methylcellulose 0.04 % p/v and saturated ammonium sulphate solution. Absorbance measurements at 280 nm were compared to a water sample treated in the same way. The difference between both solutions indicated the concentration of total tannins (Sarneckis *et al.* 2006).

For the determination of volatile compounds, a solid-phase extraction (SPE) method was applied before the chromatographic analysis on a GC-MS system (model GCMS-TQ8040) using the method of Piñeiro *et al.* 2004. The GC column was a SupraWax-280 column (60 m x 0.25 mm x 0.25 µm). The gradient conditions were 50 °C (2 min), up to 250 °C at a rate of 10 $°C \cdot min^{-1}$. Finally, the temperature was increased up to 270 °C (2 min) at a rate of 40 $°C \cdot min^{-1}$. Analysis were run five months after bottling the wines.

The peaks were identified by means of the NIST 1.4 library (Gainthersburg, EEUU) by analogy with the mass spectra and then, confirmed based on the standards reten-

tion rates when possible, or according to the retention data found in the literature (Selli *et al.* 2003 and 2006, Guerrero *et al.* 2007, Zhang *et al.* 2015, Baron *et al.* 2016).

For the quantification of the compounds of the extracts made by the SPE technique, the recovery of the analyte was studied, since by means of the SPE technique the entire analyte is not recovered. Standard samples of aromatic compounds with known concentrations in a matrix of ethanol: 12 % water and also in another wine matrix were made. These samples were processed by the SPE technique and the extracts were analyzed in the GC-MS system. The recovery percentage was calculated by comparison between the result in the GC-MS system and the starting value in the sample.

Descriptive sensory analysis: The six wines were evaluated five months after bottling by a trained panel of 12 volunteers formed by seven women and five men ranging from 21 to 51 years of age. All the panelists had wine tasting experience and were selected based on their availability and interest. Four training sessions were held to allow terminology and reference standards to be agreed upon Lenoir and Raymond (2000). Following the training, panelists were asked to evaluate the six wines in three tasting sessions. In each session, six random wines were tasted. Four attributes were evaluated always in the same order: red fruit, floral, pepper and astringency texture (Anzaldúa Morales 1994).

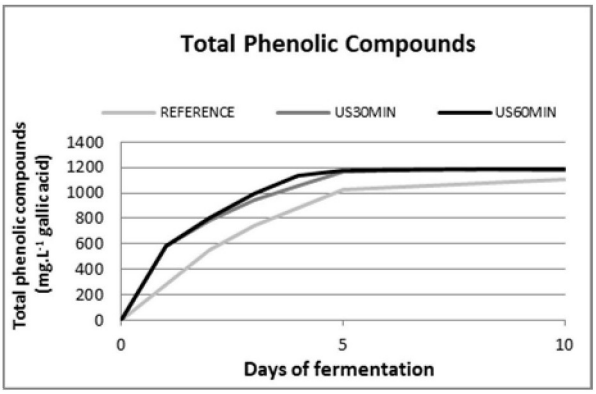
Results and Discussion

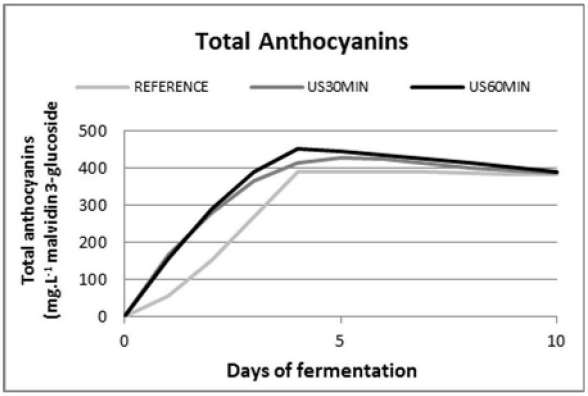
The musts and wines from the three winemaking conditions were evaluated for their levels of routine parameters, phenolic compounds, volatile compound contents and tasting results.

Fig. 2 (A) shows the evolution of the total phenolic compounds for the three different fermentation conditions assayed. It can be seen that from the beginning, the levels of total phenolics were higher in the musts treated with ultrasounds. The highest difference was obtained after 2 d of treatment. Values +41 % for US30MIN and +45 % for US 60 MIN higher than the reference must were registered for total phenolic compounds. Similar results have already been described on the effects of ultrasound by applying it to food (Chemat and Khan 2011) to red grape residues (Usaquén-Castro *et al.* 2006) or to wine fermentation (El Darra *et al.* 2013).

However, upon the fermentation had been completed, no significant differences were found among the final levels for phenolic compounds in the three resulting wines. The Table shows the results corresponding to total phenolic compounds in three final wines: 1170 mg·L^{-1} of total phenolics (gallic acid equivalents) in the REFERENCE wine versus 1212 mg·L^{-1} of total phenolics in the US60MIN wine.

Fig. 2 (B) shows total anthocyanin contents. Differences for total anthocyanins between the reference must (REFERENCE) and the musts obtained using ultrasounds after the second day of the alcoholic fermentation, were greater than in phenolic compounds, with +87 % for US30





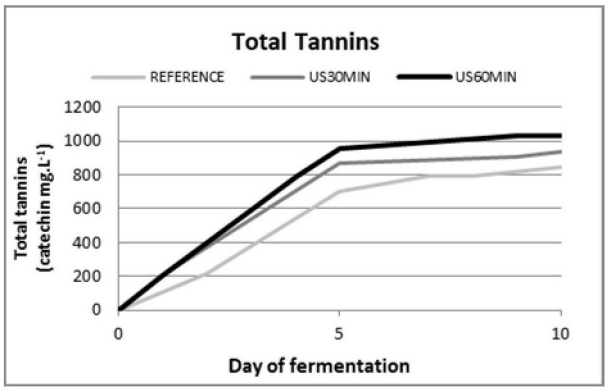


Fig. 2: Evolution of the extraction of total anthocyanins (**A**), total phenolic compounds (**B**) and total tannins (**C**) during alcoholic fermentation.

MIN and +93 % for US 60 MIN in comparison with the reference must (REFERENCE). These results coincide with other studies on anthocyanin extraction by applying ultrasound in grape by-products (Corrales *et al.* 2008) or plum and grape peels (Liu 2016). Nevertheless, after 16 d of application, the levels for total anthocyanins in the resulting wines showed non-significant differences at 95 % level of confidence (Table). Reference 'Syrah' wines reached 303 mg·L^{-1} of total anthocyanins (expressed as malvidin-3-glucoside), whilst US60MIN wines reached 316 mg·L^{-1} total anthocyanins content. Fig. 2 (C) shows the evolution during the fermentation of the total tannin contents. From the beginning, the total tannin contents were higher in those musts that had been treated with ultrasound. The biggest differences were registered after 5 d,

Table

Absorbance at 520 nm, total polyphenols, total anthocyanins, total tannins and ethanol measured (average of 6 determinations (3 analyses in 2 different wines ± standard deviation) in the final wines (30 d after the alcoholic fermentation). Within the same column, values bearing different superscript letters differ significantly ($p < 0.05$)

	Absorbance at 520 nm	Total polyphenols ($mg \cdot L^{-1}$)	Total anthocyanins ($mg \cdot L^{-1}$)	Total tannins ($mg \cdot L^{-1}$)	Ethanol (%)
Reference	0.581 ± 0.02^{a}	1170 ± 19.2^{a}	303 ± 12.3^{a}	1100.8 ± 31.2^{a}	12.60 ± 0.15^{a}
US30MIN	0.610 ± 0.01^{b}	1185 ± 18.8^{a}	303 ± 22.2^{a}	$1178.2 \pm 20.8^{a,b}$	12.54 ± 0.11^{a}
US60MIN	0.637 ± 0.01^{c}	1212 ± 16.1^{a}	316 ± 10.6^{a}	1230.9 ± 32.3^{b}	12.56 ± 0.18^{a}

(+ 23 % for US30MIN and + 36 % for US 60 MIN). After the fermentation, the resulting differences for total tannins between the wines obtained with ultrasound and the reference wines were largely reduced. Only the wine obtained using the longest treatment with ultrasound (US60MIN) showed a significantly higher value for total tannins than the reference wines (1231 vs 1101 $mg \cdot L^{-1}$ of total tannins content expressed as catechin).

Therefore, it can be concluded that the application of ultrasounds speeds the extraction processes up from the grape skins and seeds. However, due to the long extraction period (16 d), similar extraction yields were found for the three resulting wines.

The effects of the winemaking techniques applied were also evaluated with regards to the aromatic composition of the final wines. The final aroma of a wine comes from both the grape and the fermentation process and it is the aromatic precursors of the grape (terpenic compounds, aldehydes, alcohols, C13 norisoprenoids and phenolic esters) which give the varietal character to the wine (Kennedy *et al.* 2000, Ferreira *et al.* 2002).

These aroma precursors are found mostly in the solid parts of the grape. In the elaboration of a red wine, the skin is in contact with the must, during the whole fermentation period, favoring this diffusion to the wine. In this regard there is an extensive bibliography (Selli *et al.* 2006, Mihnea *et al.* 2015, Ferreira *et al.* 2000). In order to study the effects of ultrasound treatment on wines, the research focused on those aroma components of interest that are in high concentrations and can be easily quantified.

The compounds chosen are those whose recovery in the analysis method reaches at least 85 %. This ensures the reliability of the integration of chromatographic peaks.

Fig. 3 shows the relative values of volatile compounds in treated wines vs their content in the reference wines. For that purpose, a value of 100 was assigned to the level of each volatile compound in the reference wines. Values above 100 % means higher values than in the reference wines.

It can be seen that all the identified compounds showed much higher levels in those wines obtained when ultrasounds had been applied according to either assayed conditions, *i.e.* 30 min or 60 min. Significant differences ($p < 0.05$) were found for all compounds in Fig. 3. To discuss the results of aroma compounds were grouped according to their nature:

- Terpenic compounds, nerol and geraniol, two compounds that are generally associated with flowery aroma

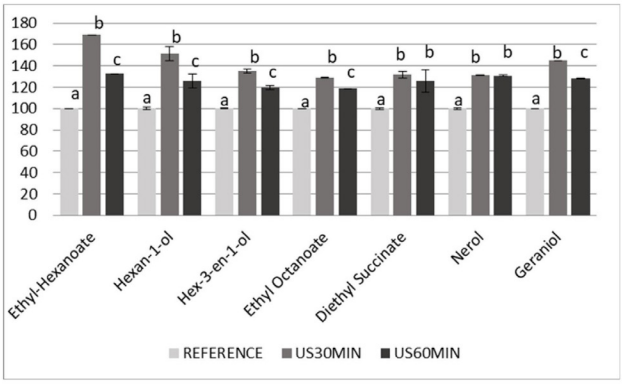


Fig. 3: Relative levels of volatile compounds in final wines. Levels in the Reference wines were fixed to 100 %. For the same compound, bars bearing different letters differ significantly ($p < 0.05$).

(Zhang *et al.* 2015, Mayr *et al.* 2014, Wang *et al.* 2016). They can be found mainly in the grape skin, therefore higher levels in wines should be due to a better extraction efficiency during the fermentation process in red wines. Nerol and geraniol exhibited a 30 % higher level in the wines produced using ultrasound during the alcoholic fermentation, than their reference wine produced by traditional fermentation procedures. Therefore, ultrasound allows for a higher extraction recovery from the grape skins related to these specific compounds.

- Alcohols, specifically, hydroxyl compounds, including hex-3-en-1-ol and hexan-1-ol. They belong to the so-called C6-compounds. These compounds are formed in grapes during pre-fermentative steps including harvesting, crushing and grape maceration. They derive from membrane lipids, *via* the formation of hexanal from linoleic and α-linolenic acids (Oliveira *et al.* 2006). These compounds showed between 20 and 60 % higher contents in both wines treated with ultrasound. Therefore, their levels also demonstrate the higher extraction efficiency by the ultrasounds during the alcoholic fermentation.

- Ethyl esters, ethyl hexanoate, ethyl octanoate, diethyl succinate, are formed during alcoholic fermentation by the esterification of fatty acids with ethanol. The acetates of higher alcohols and the ethyl esters of fatty acids are associated whit floral and fruity aromas in young wines. Linear (C2-C4), medium (C6-C10), long (C6-C10) and branched (2-methyl propanoic, 2-methyl butanoic, etc.) volatile fatty acids are produced during alcoholic fermentation (Francis and Newton 2005). The wines that had been produced

using ultrasound showed up to 70 % increased levels of esters *vs* the reference wine. Ethyl hexanoate (pineapple flavour, Mayr 2014) increases by 69 % in US30MIN wine and 33 % in US60MIN wine. Ethyl octanoate (melon related aroma, Mayr 2014) increases by 29 % in US 30MIN and by 19 % in US60MIN. Diethyl succinate is an aromatic ester (also melon related aroma, Peinado, 2004) that increased its concentration by 69 % in US30MIN and 33 % in US60MIN.

Therefore, in some cases, longer ultrasound applications produced lower levels of some aroma related compounds. This means that some degree of degradation took place when ultrasound treatments was prolonged.

Final levels for ethyl esters are directly related to the yeast metabolism and the effect of ultrasound application (Koda *et al.* 2009, Kulkarni *et al.* 2015). Assuming that greater amounts of alcohols come from the de-amination of amino acids (Piñeiro *et al.* 2006), those metabolic routes that need greater amounts of nitrogen would increase their activity (El-Dalatony *et al.* 2019). The evolution of the fermentation was then checked. It must be noted that after 42 h of fermentation, there were differences between ultrasound treated must and reference one. Reference musts had a density of 1.0553 ± 0.001 ($g \cdot mL^{-1}$) higher than densities of US30MIN (1.0487 ± 0.001) and US60MIN (1.0455 ± 0,001) musts. Those differences remained stable over the rest of the fermentation time. Therefore, the fermentation process with ultrasound was faster than the alcoholic fermentation in the reference must. In this way, the application of ultrasound during the fermentation phase may affect the composition of the fatty acids in must due to changes in fermentation rates. It has been described that under anaerobic conditions, yeast produces medium-chain fatty acids, while when fermentation is carried out in aerobic or semi-aerobic conditions, more unsaturated fatty acids are produced (Torija *et al.* 2003). So, the effects of ultrasounds could be related to the level of oxygen in must during the alcoholic fermentation.

In conclusion, the final levels in the wines that had been produced by ultrasound treatments were between 20 % and 60 % higher than those of the reference wines (REFERENCE). The use of ultrasound during the fermentation process did not reduce the volatile contents as a result of their degradation or for any other reason, but ultrasound treatments allowed for the production of more aromatic wines.

Tasting panel: In the sensory analysis, the six wines were evaluated by a panel of trained volunteers. Discriminatory tests were carried out in accordance with the UNE-EN ISO 4120 Standard (AENOR 2010) to determine if the wines resulting from the treatments were different. The results showed that the wines resulting from the study were different with a degree of significance of 99.9 %. The panelists were able to discriminate the reference wine in 91 % of the cases. They could also distinguish USMIN30 and USMIN60 wines in 75 and 86 % of the cases respectively.

On the other hand, Fig. 4 shows the results of the descriptive analysis. US30MIN wine had the highest score for fruit aromas (+11 % compared to the Reference wine) and flower aromas (+36 % compared to the Reference wine). The highest astringency intensity was reported for the wine with the longest ultrasound treatment, *i.e.* US-60MIN (+ 31 % compared to the Reference wine). These tasting results are supported by the results previously found for terpenic compounds and total tannins. Levels for compounds such as nerol or geraniol provideing flowery aromas (Wang *et al.* 2016) were higher in must obtained using ultrasound. Additionally, those wines also showed higher levels for total tannins, then producing an stronger astringency.

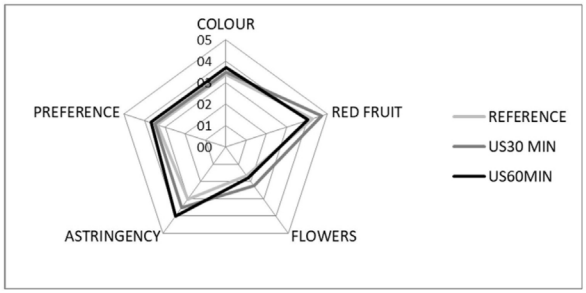


Fig. 4: Spider plot of descriptive analysis attributes for the three final wines (average of duplicate wines): Reference, US30MIN and US60MIN.

Conclusions

Ultrasound applied to red grape musts during its alcoholic fermentation allows for additional extraction of some specific compounds from the solid parts in grapes, including some terpenic compounds. Additionally, ultrasound speeds up the fermentative process, which modifies the metabolism of grapes. It produced changes in some related compounds: ethyl esters. Ultrasound also speed up the extraction process of phenolic related compounds. However, the final content levels were not different for total phenolics and total anthocyanins, since similar amounts of those compounds were finally extracted by applying regular winemaking procedures. Final flavour properties for wines with and without ultrasound during the alcoholic fermentation were different. Ultrasound allowed for more aromatic wines, also with increased astringency.

References

AENOR; 2010: Análisis Sensorial, Metodología, Ordenación. UNE-EN ISO 8587:2010, Madrid, Spain.

Álvarez, I.; Aleixandre, J. L.; García, M. J.; Lizama, V.; Aleixandre-Tudó, J. L.; 2009: Effect of the prefermentative addition of copigments on the polyphenolic composition of Tempranillo wines after malolactic fermentation. Eur. Food Res. Technol. **228**, 501-510.

Anzaldúa Morales, A.; 1994: La Evaluación Sensorial de los Alimentos en Teoría y la Práctica. Acrabia.

Baron, M.; Sochor, J.; Prusova, B.; Tomaskova, L.; Kumsta, M.; 2016: A study on the content of terpenic compounds in the cultivar 'Moravian muscat' (*Vitis vinifera* L.). Italian J. Food Sci. **29**, 222-232.

Bosso, A.; Guaita, M.; Panero, L.; Borsa, D.; Follis, R.; 2009: Influence of two winemaking techniques on polyphenolic composition and color of wines. Am. J. Enol. Vitic. **60**, 379-385.

Busse-Valverde, N.; Gómez-Plaza, E.; López-Roca, J. M.; Gil-Muñoz, R.; Fernández-Fernández, J. I.; Bautista-Ortín, A. B.; 2010: Effect

of different enological practices on skin and seed proanthocyanidins in three varietal wines. J. Agric. Food Chem. **58**, 11333-11339.

Carrera, C.; Ruiz-Rodríguez, A.; Palma, M.; Barroso, C. G.; 2012: Ultrasound assisted extraction of phenolic compounds from grapes. Analyt. Chim. Acta **732**, 100-104.

Casassa, L. F.; Bolcato, E. A.; Sari, S. E.; 2015: Chemical, chromatic, and sensory attributes of 6 red wines produced with prefermentative cold soak. Food Chem. **174**, 110-118.

Cejudo-Bastante, M. J.; Gordillo, B.; Hernanz, D.; Escudero-Gilete, M. L.; González-Miret, M. L.; Heredia, F. J.; 2014: Effect of the time of cold maceration on the evolution of phenolic compounds and colour of Syrah wines elaborated in warm climate. Int. J. Food Sci. Technol. **49**, 1886-1892.

Chang, A. C.; 2005: Study of ultrasonic wave treatments for accelerating the aging process in a rice alcoholic beverage. Food Chem. **92**, 337-342.

Chemat, F.; Khan M. K.; 2011: Applications of ultrasound in food technology: processing, preservation and extraction. Ultrason. Sonochem. **18**, 813-835.

Corrales, M.; Toepfl, S.; Butz, P.; Knorr, D.; Tauscher, B.; 2008: Extraction of anthocyanins from grape by-products assisted by ultrasonics, high hydrostatic pressure or pulsed electric fields: A comparison. Innovat. Food Sci. Emerging Technol. **9**, 85-91.

El-Dalatony, M. M.; Saha, S.; Govindwar, S. P.; Abou-Shanab, R. A.; Jeon, B.-H.; 2019: Biological conversion of amino acids to higher alcohols. Trends Biotechnol. **37**, 855-869.

El Darra, N.; Grimi, N.; Maroun, R. G.; Louka, N.; Vorobiev, E.; 2013: Pulsed electric field, ultrasound, and thermal pretreatments for better phenolic extraction during red fermentation. Eur. Food Res. Technol. **236**, 47-56.

Ferraretto, P.; Celotti, E.; 2016: Preliminary study of the effects of ultrasound on red wine polyphenols. CyTA-J. Food **14**, 529-535.

Ferreira, V.; Ortín, N.; Escudero, A.; López, R.; Cacho, J.; 2002: Chemical characterization of the aroma of Grenache rose wines: Aroma extract dilution analysis, quantitative determination, and sensory reconstitution studies. J. Agric. Food Chem. **50**, 4048-4054.

Ferreira, V.; López, R.; Cacho, J. F.; 2000: Quantitative determination of the odorants of young red wines from different grape varieties. J. Sci. Food Agric. **80**, 1659-1667.

Francis, I.; Newton, J.; 2005: Determining wine aroma from compositional data. Aust. J. Grape Wine Res. **11**, 114-126.

Gishen, M.; Dambergs R.; Cozzolino D.; 2005: Grape and wine analysis-enhancing the power of spectroscopy with chemometrics. A review of some applications in the Australian wine industry. Aust. J. Grape Wine Res. **11**, 296-305.

González-Neves, G.; Gil, G.; Favre, G.; Ferrer, M.; 2012: Influence of grape composition and winemaking on the anthocyanin composition of red wines of Tannat. Int. J. Food Sci. Technol. **47**, 900-909.

Guerrero, E. D.; Marín, R. N.; Mejías, R. C.; Barroso, C. G.; 2007: Stir bar sorptive extraction of volatile compounds in vinegar: Validation study and comparison with solid phase microextraction. J. Chromatog. A, **1167**, 18-26.

Hernanz, D.; Gallo, V.; Recamales, A. F.; Meléndez-Martínez, A. J.; Heredia, F. J.; 2008: Comparison of the effectiveness of solid-phase and ultrasound-mediated liquid-liquid extractions to determine the volatile compounds of wine. Talanta **76**, 929-935.

Kennedy, J. A.; Matthews M. A.; Waterhouse A. L.; 2000: Changes in grape seed polyphenols during fruit ripening. Phytochemistry **55**, 77-85.

Koda, S.; Miyamoto, M.; Toma, M.; Matsuoka, T.; Maebayashi, M.; 2009: Inactivation of *Escherichia coli* and *Streptococcus mutans* by ultrasound at 500 kHz. Ultrason. Sonochem. **16**, 655-659.

Kulkarni, P.; Loira, I;, Morata, A.; Tesfaye, W.; González, M. C.; Suárez-Lepe, J. A.; 2015: Use of non-*Saccharomyces* yeast strains coupled with ultrasound treatment as a novel technique to accelerate ageing on lees of red wines and its repercussion in sensorial parameters. LWT-Food Sci. Technol. **64**, 1255-1262.

Lenoir, J.; Raymond, R.; 2000: Le Nez du Vin. Éditions Jean Lenoir, Carnoux-en-Provence, France.

Liu, L.; Loira, I.; Morata, A.; Suárez-Lepe, J.; González, M.; Rauhut, D.; 2016: Shortening the ageing on lees process in wines by using ultrasound and microwave treatments both combined with stirring and abrasion techniques. Eur. Food Res. Technol. **242**, 559-569.

Mayr, C. M.; Geue, J. P.; Holt, H. E.; Pearson, W. P.; Jeffery, D. W.; Francis, I. L.; 2014: Characterization of the key aroma compounds in Shiraz wine by quantitation, aroma reconstitution, and omission studies. J. Agric. Food Chem. **62**, 4528-4536.

Mazza, K. E.; Santiago, M. C.; do Nascimento, L. S.; Godoy, R. L.; Souza, E. F.; Brígida, A. I. S.; Tonon, R. V.;2018: Syrah grape skin valorisation using ultrasound-assisted extraction: Phenolic compounds recovery, antioxidant capacity and phenolic profile. Int. J. Food Sci. Technol. **54**, 641-650.

Mihnea, M.; González-SanJosé, M.; Ortega-Heras, M.; Pérez-Magariño, S.; 2015: A comparative study of the volatile content of Mencía wines obtained using different pre-fermentative maceration techniques. LWT-Food Sci. Technol. **64**, 32-41.

Oliveira, J. M.; Faria, M., Sá, F.; Barros, F.; Araújo, I. M.; 2006: C6-alcohols as varietal markers for assessment of wine origin. Analyt. Chim. Acta **563**, 300-309.

Panprivech, S.; Lerno, L. A.; Brenneman, C. A.; Block, D. E.; Oberholster, A.; 2015: Investigating the effect of cold soak duration on phenolic extraction during Cabernet Sauvignon fermentation. Molecules **20**, 7974-7989.

Peinado, R. A.; Moreno, J.; Bueno, J. E. ; Moreno, J. A.; Mauricio, J. C.;2004: Comparative study of aromatic compounds in two young white wines subjected to pre-fermentative cryomaceration. Food Chem. **84**, 585-590.

Piñeiro, Z.; Natera, R.; Castro, R.; Palma, M; Puertas, B.; Barroso, C.; 2006: Characterisation of volatile fraction of monovarietal wines: Influence of winemaking practices. Analyt. Chim. Acta **563**, 165-172.

Piñeiro, Z.; Palma M.; Barroso C. G.; 2004: Determination of terpenoids in wines by solid phase extraction and gas chromatography. Analyt. Chim. Acta **513**, 209-214.

Sacchi, K. L.; Bisson, L. F.; Adams, D. O.; 2005: A review of the effect of winemaking techniques on phenolic extraction in red wines. Am. J. Enol. Vitic. **56**, 197-206.

Sarneckis, C. J.; Dambergs, R.; Jones, P.; Mercurio, M.; Herderich, M. J.; Smith, P.; 2006: Quantification of condensed tannins by precipitation with methyl cellulose: development and validation of an optimised tool for grape and wine analysis. Aust. J. Grape Wine Res. **12**, 39-49.

Selli, S.; Cabaroglu, T.; Canbas, A.; Erten, H.; Nurgel, C.; 2003: Effect of skin contact on the aroma composition of the musts of *Vitis vinifera* L. cv. Muscat of Bornova and Narince grown in Turkey. Food Chem. **81**, 341-347.

Selli, S.; Canbas, A.; Cabaroglu, T.; Erten, H.; Günata, S.; 2006: Aroma components of cv. Muscat of Bornova wines and influence of skin contact treatment. Food Chem. **94**, 319-326.

Torija, M. a. J.; Beltran, G.; Novo, M.; Poblet, M.; Guillamón, J. M.; Mas, A.; Rozès, N.; 2003: Effects of fermentation temperature and *Saccharomyces* species on the cell fatty acid composition and presence of volatile compounds in wine. Int. J. Food Microbiol. **85**, 127-136.

Usaquén-Castro, X.; Martínez-Rubio, M.; Aya-Baquero, H.; González-Martínez, G.; 2006: Ultrasound-assisted extraction of polyphenols from red-grape (*Vitis vinifera*) residues, 1315-1324. IUFoST 13th World Congr. Food Sci. Technol., 17-21 September 2006, Nantes, France.

Vila, D. H.; Mira, F. J. H.; Lucena, R. B.; Recamales, M. F.; 1999: Optimization of an extraction method of aroma compounds in white wine using ultrasound. Talanta **50**, 413-421.

Wang, L.; Hu, G.; Lei, L.; Lin, L.; Wang, D.; Wu, J.; 2016: Identification and aroma impact of volatile terpenes in Moutai liquor. Int. J. Food Propert. **19**, 1335-1352.

Zhang, S.; Petersen, M. A.; Liu, J.; Toldam-Andersen, T. B.;.2015: Influence of pre-fermentation treatments on wine volatile and sensory profile of the new disease tolerant cultivar Solaris. Molecules **20**, 21609-21625.

Zhang, Q. A.; Wang, T. T.; 2017: Effect of ultrasound irradiation on the evolution of color properties and major phenolic compounds in wine during storage. Food Chem. **234**, 372-380.

Zheng, X.; Zhang, M.; Fang, Z., Liu, Y.; 2014: Effects of low frequency ultrasonic treatment on the maturation of steeped greengage wine. Food Chem. **162**, 264-269.

Received September 12, 2019