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EFFECTS OF WINEMAKING ON ‘MARSELAN’ RED WINES: VOLATILE COMPOUNDS AND SENSORY ASPECTS

EFEITOS DE DIFERENTES PROCESSOS DE VINIFICAÇÃO EM VINHOS TINTOS ‘MARSELAN’: COMPOSTOS VOLÁTEIS E ASPECTOS SENSORIAIS

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SUMMARY

Winemaking processes and volatile compounds perform an important role in contributing to the quality of wines. ‘Marselan’ is a red grape variety grown in several countries such as France, Brazil and China. This variety has gained international interest in recent years. The volatile profile of Brazilian ‘Marselan’ red wines during various winemaking processes was studied. Four processes were applied: thermovinification, *integrale* vinification, classic winemaking and classic winemaking pretreated with ultrasound. The wines were characterized with conventional physicochemical analyses, volatile composition and sensory analysis. Sixty-three volatile compounds were identified and semi-quantified. The wines presented different sensory characteristics. The thermovinification and classic winemaking with ultrasound application methods showed a large presence of esters contributing to a fruity and overmaturation aroma in the volatile profile and sensory analysis. An increased presence of vegetal and sulfurous aromas was observed in the wines from the classic winemaking and *integrale* vinification processes. This effect was most likely attributed to the lower influence of esters which generated a greater perception of other compounds and conferred these aromas. The differences can be directly linked to the various extraction rates in the vinification processes, which result in different interactions between the compounds.

RESUMO

Os processos de vinificação e a composição volátil são fatores muito importantes para a qualidade dos vinhos. ‘Marselan’ é uma casta tinta cultivada em diversos países, como França, Brasil e China. Além disso, esta variedade vem ganhando maior interesse internacional nos últimos anos. Neste trabalho, foi estudado o perfil volátil de vinhos oriundos da uva ‘Marselan’, submetidos a diferentes processos de vinificação. Foram aplicados quatro processos: termovinificação, vinificação integral, vinificação clássica e vinificação clássica com aplicação de ultrassom. Os vinhos foram caracterizados por análises físico-químicas, composição volátil e análise sensorial. Sessenta e três compostos voláteis foram identificados e semi-quantificados. Os vinhos apresentaram diferentes características sensoriais. A composição volátil mostrou uma grande presença de ésteres, que contribuíram para um aroma frutado e de sobrematuração nos processos de termovinificação e vinificação clássica com aplicação de ultrassom. A presença de aromas vegetais e sulfurosos foi observada nos processos de vinificação clássica e vinificação integral. Este efeito provavelmente ocorreu devido à menor influência de ésteres, gerando maior percepção de outros compostos que conferem estes aromas. As diferenças podem estar diretamente ligadas às diferentes taxas de extração nos processos de vinificação, que subsequentemente resultam em diferentes interações entre os compostos.

Key words: Volatile profile, thermovinification, *integrale* vinification, ultrasound application, Marselan wine quality.

Palavras-chave: Perfil volátil, termovinificação, vinificação integral, aplicação de ultrassom, qualidade do vinho Marselan.

INTRODUCTION

Wine is a complex alcoholic beverage formed by several compounds. Aroma compounds are one of the most important groups to determine the level of quality in a wine (Villamor and Ross, 2013; Fariña *et al.*, 2015). The grape variety and the technology used during winemaking are crucial for odor quality in wines (Geffroy *et al.*, 2015; Ostapenko *et al.*, 2017; De Castilhos *et al.*, 2019). ‘Marselan’ is a red grape variety that resulted of the crossing of ‘Cabernet Sauvignon’ and ‘Grenache’ grapevine varieties in Marseille, France. The wine presents a high degree of phenolic compounds, intense color and good tannic structure (Robinson *et al.*, 2013; INRA, 2020). Due to its sensory characteristics, ‘Marselan’ has become of great importance in new world wine countries, such as China, which has the most ‘Marselan’ plantings in the world (Ma, 2017; Jiao and Oujiang, 2019; Lyu *et al.*, 2019). Historically, the wine market has expanded when unfamiliar grape varieties are associated with non-traditional elaboration processes (Ostapenko *et al.*, 2017).

Wine aromas from the ‘Marselan’ grape consist of black fruits, spices, cocoa and vegetal characteristics (INRA, 2020). The latter is due to the crossing of ‘Cabernet Sauvignon’ and ‘Grenache’ (Miele and Rizzon, 2011). The study of volatile composition of ‘Marselan’ wines was found in two scientific articles. Lyu *et al.* (2019) characterized the key aromatic compounds in ‘Marselan’ wines. The researchers found 43 odor-active compounds. The aroma profile can be described with notes such as blackberry, green pepper, honey, raspberry, caramel, smoke, and cinnamon.

Ostapenko *et al.* (2017) studied the sensory and chemical attributes of dessert wines created by various freezing methods of ‘Marselan’ grapes. The aroma profile can be characterized as fruity (pear, plum and apricots), citrus, nutty tones, with hints of spice, caramel and honey.

The winemaking process is a crucial step to define the quality of wines. It has a major influence on the main volatile compounds and their precursors. Factors such as maceration time, extraction rates, hydrolysis and oxidation influence the aromatic characteristics of wines (Guerra, 2002), and help to create distinct wines in the market. In classic winemaking, the must is collected by destemming and crushing the grapes (Baiano *et al.*, 2016). The must remains in contact with the skins and seeds for an extended period of time in stainless steel tanks. The time allotted for this maceration will depend on the quality of the raw material and desired characteristics for the final wine (Guerra, 2002; Baiano *et al.*, 2016).

During the *integrale* vinification method, the must and solid parts are placed in oak barrels, where the vinification process occurs. This method promotes greater harmony between wine and wood (Gagliole, 2013). *Integrale* vinification gives greater volume and depth to wines by the smooth extraction of compounds through prolonged contact with alcohol in small barrels (Gagliole, 2013).

Thermovinification is the basic principle of heating grapes at temperatures close to boiling. Once the grapes are destemmed and crushed, they are heated to temperatures between 80 °C and 85 °C, then cooled to 35 °C or 40 °C. This sudden temperature shock increases the extraction of compounds (Wang *et al.*, 2016). The effectiveness of the process depends on the temperature regulation and grape variety. Some varieties are better adapted for thermovinification than others (Baiano *et al.*, 2016).

The ultrasound technique uses ultrasonic energy on the grapes. This causes the physical effect of acoustic cavitation; the formation, collapsing and explosion of micro bubbles (100 µm) in a localized hotspot. Acoustic cavitation produces a high amount of energy and pressure, generating shock waves, which cause cellular rupture. Due to the acoustic transmission, an increase of mass transfer rates occurs, increasing the diffusion of the compounds (Mason, 1998; Toma *et al.*, 2001; Ferraretto and Celotti, 2016).

Some studies show analysis of red wines made by different technologies (Mihnea *et al.*, 2014; Geffroy *et al.*, 2015; Ostapenko *et al.*, 2017; De Castilhos *et al.*, 2019). However, there are no studies analyzing ‘Marselan’ red winemaking with the aforementioned processes. Due to the lack of research, this work is focused on the application of different winemaking processes, such as *integrale* vinification (IV), thermovinification (TV), classic winemaking (CW) and classic winemaking pretreated with ultrasound application (CWUS) to examine the influence on volatile composition and sensorial quality on ‘Marselan’ red wines. The research questions are, “Do different winemaking processes cause change in the volatile composition of wines? If so, what would be the best winemaking process?”.

MATERIAL AND METHODS

Samples

Grapes: Fresh, healthy, and ripe (°Brix between 23 and 25) ‘Marselan’ (*Vitis vinifera L.*) grapes were obtained from Villaggio Grando Winery, located in Água Doce city, Santa Catarina, Brazil (Latitude 26°43’31.73’’S; Longitude 51°30’35.699’’W; Altitude 1250.22 m). The harvest was completed in

March of 2018. The harvested grapes were immediately transported from the vineyard to Embrapa in refrigerated vehicles. The grape bunches had an average weight of 100 g to 150 g.

Vineyard characteristics: i) guyot pruning method (remaining number of nodes per vine after pruning was between 6 and 8); ii) elevation – first trellis wire 1.10 m; second trellis wire 1.53 m; iii) canopy management practices - shoot topping performed 2 or 3 times per year; iv) pest management program - treatments only when raining (as required); v) fertilization program – annual limestone additions depending on soil analysis (1000 kg of limestone per hectare) the required amount of NPK fertilizers is applied and no irrigation system is used; vi) vine slope - 100% absolute North-South; vii) soil type - oxisol with an average depth of 7 m; viii) rootstock variety - Paulsen 1104; ix) vine spacing - 1.1 m between plants and 2.8 m between rows; x) timing of key phenological stages – the pruning occurs between June and July and harvest occurs between 20 February and 20 April; xi) average age of the vines – 22 years.

Climate conditions: i) average rainfall - 1433 mm; ii) annual sunshine - from 2045 h to 2523 h; iii) relative humidity - 77.3%; iv) average temperature - 14.6 °C.

Winemaking processes

Wines were elaborated in the Embrapa Grape and Wine Research Center, located in the city of Bento Gonçalves, Rio Grande do Sul, Brazil. Four winemaking processes were applied to ‘Marselan’ grapes during the 2018 harvest; exclusive of any impact from vintage variations since the aim of this work is to understand the impact of the winemaking processes. Four treatments were prepared; as a result of one properly controlled vinification per treatment. The vinifications were conducted by experienced winemakers and all analyses were performed three times:

i) Classic winemaking (CW): 21 kg of grapes were destemmed and crushed (destemmer - crushing Enoveneta - model 40) and pressed (Ricefer - specially designed for small volumes) in a stainless steel tank and macerated for 10 days at 25 °C. After this stage, the “drawing off” phase was completed and the fermentation began. Active dry yeast was used in the form of *Saccharomyces cerevisiae* (RX-60 Lallemand) at a rate of 30 g/hL. The yeast was diluted in a must aliquot for each vinification process and added to each fermentation vessel. Homogenization took place in a climate controlled room at 20 °C for 10 days. No fermentation activator or pectinolytic enzymes

were added to the must. The initial concentration of SO₂ was 80 mg/L. After the fermentation, a cold stabilization lasted for 15 days at 5 °C. All winemaking process showed an average yield of 60%.

- ii) *Integrale* vinification (IV): 76 kg of ‘Marselan’ grapes were destemmed, crushed (the same process as in classic winemaking) and macerated for 10 days at 25 °C in previously used 12 month old oak barrels (*Quercus robur L.*). The fermentation time was 10 days. A total of 50 L was processed. After maceration, the “drawing off” phase was completed. The fermentation continued in a stainless steel tank located in a climate controlled room at 20 °C. The remaining steps in the process were the same as the Classic Winemaking method.
- iii) Thermovinification (TV): 76 kg of grapes were destemmed and crushed (the same process as in classic winemaking). The grapes were divided into two batches of equal volume and processed the same way (Suquificador Integral SI 70 kg - BR 10 2016002718-7) as to not overload the equipment. The grapes were placed in the equipment and heated to 90 °C. Then, the must was transferred to stainless steel tanks, and cooled to 20 °C. The duration of the fermentation period was 7 days. A total of 50 L was processed. The remaining steps of the process were the same as the Classic Winemaking method.
- iv) Classic winemaking with ultrasound application (CWUS): After the grapes were destemmed and crushed – 25 kg – (the same process as in classic winemaking), the solid mass and part of the liquid (approximately 8 L of liquid was removed to decrease the number of batches) were subjected to ultrasonic treatment in an ultrasound bath for 30 minutes at 25 °C in 2 batches of 8.5 kg each (Thornton®, model T50; Power: 127 W; Frequency: 40 kHz; Dimensions: 30 cm x 50 cm x 20 cm). The remaining steps of the process were the same as the Classic Winemaking method.

All the wines were bottled in 375 mL bottles, labeled and stored in a climate controlled room regulated to 18 °C until analysis was completed one year later. All other variables were properly controlled.

Physicochemical analyses

The oenological parameters were analyzed following the OIV methodology (OIV, 2019). The density was measured by densimetry. Total acidity was determined by potentiometric titration, and was expressed as g/L of tartaric acid. Volatile acids were separated from the wine by steam distillation and

titrated using standard sodium hydroxide; the result was expressed in g/L of acetic acid. The alcoholic strength by volume was measured through distillation. The pH level was determined by potentiometry. The total dry extract was calculated indirectly from the specific gravity of the alcohol-free wine. Sugar levels were reduced by measuring the reaction of the clarified wine with a specified quantity of an alkaline copper salt solution; the excess copper ions were then determined iodometrically. Free and total sulfur dioxide was measured by titrimetry.

Volatile compounds analysis

The analysis followed the protocol described by Bernardi *et al.* (2014) with some modifications. Extraction of volatile organic compounds (VOCs) of 'Marselan' red wines was carried out by HS-SPME; using a fiber divinylbenzene/carboxen/polydimethylsiloxane (DVB/Car/PDMS, 50/30 $\mu\text{m} \times 20$ mm) under the following conditions: sample at 35 °C for 45 minutes with continuous stirring (500 rpm) with 30% of NaCl. The fiber was purchased from Supelco (Bellefonte, PA, USA). Before its first use, the fiber was thermally conditioned in accordance with manufacturer recommendations. In all of the experiments, a 2 mL aliquot of wine was transferred to a 20 mL glass vial and the extraction was performed while constantly being stirred. Before each analysis, the mixture was carefully shaken to dissolve the salt and left to equilibrate for 5 minutes.

Volatile compounds were desorbed from the fiber and moved into a split injector (Varian 3400CX gas chromatography equipped with flame ionization detector - GC/FID) at 230 °C for 10 min. The injector operated in splitless mode for 1 minute with an SPME liner (0.75 mm i.d.). The temperature of the injector and FID was set to 250 °C. Hydrogen carrier gas was added at an initial flow rate of 2 mL/min (constant pressure of 69 kPa). A ZB-Wax plus capillary column was used with the dimensions of 30 m \times 0.25 mm \times 0.25 μm film thickness (Phenomenex, Palo Alto, CA, USA). The oven temperature was set to 35 °C for 2 minutes, raised to 80 °C for 2 °C/min, 150 °C for 4 °C/min, 230 °C for 8 °C/min and maintained at this temperature for 5 minutes. For semi-quantification, the VOC concentration was calculated by internal standardization, using 3-octanol (Sigma Aldrich, Germany) at a concentration of 1.03 mg/L. The response factor between internal standard and analytes was assumed as one. All analyses were carried out in triplicate and the results were expressed as $\mu\text{g/L}$ or the equivalent of IS.

Volatile compounds were identified with Shimadzu QP2010 Plus gas chromatography coupled with a

mass spectrometer (GC/MS). GC conditions were the same as those used for the GC-FID chromatographic analysis with polar column. Helium was used as a carrier gas at a flow rate of 2 mL/min. MS was operated in electron ionization (EI) mode at 70 eV, scanning from m/z 35 to 350. The volatile compounds were tentatively identified by comparing their mass spectra to commercial spectra databases (NIST 14), and by comparing their experimental retention index (RI) with the literature.

Sensorial Analysis

A variation of the Quantitative Descriptive Analysis (QDA) as the consensus training method was employed. Fourteen judges from Embrapa Grape and Wine, Brazil, tasted the wine samples three times with reference standards in two sessions. Six samples were evaluated per session with some modifications (1 hour per session) as reported by De Castilhos *et al.* (2019). All judges have experience in sensory analysis. The analysis began with a discussion regarding the attributes of the wines. The session president wrote down all of the aromatic descriptors found by the judges to generate a protocol. The generated protocol served as the basis for generating the evaluation form. Before each session, a training session was held with aromatic and flavor standards of wines available to the judges.

A discussion with the judges resulted in organizing wine attributes into seven total odor descriptors: aroma intensity, aroma multiplicity/complexity, fruit aroma, vegetal aroma, overmaturation aroma, spice aroma and undesirable aroma.

The evaluation session took place in a sensory analysis room equipped with individual cabins under daylight at ambient temperature. 40 mL aliquots of the analyzed wines were offered to the judges in a wine glass coded with three random digits in a random order. For each sample, the subject rated the intensity of these descriptors on a 90 mm electronic scale read left to right, "no odor perceived" to "very intense".

The Ethical Issues regarding the QDA was approved by the Ethics in Research Committee of the Federal University of Rio Grande do Sul (protocol n°. 25063519.5.0000.5347).

Statistical Analysis

The data from the volatile and sensory analyses was analyzed using one-way Analysis of Variance (ANOVA), a Tukey test ($p < 0.05$) using BioEstat 5.3, and a Principal Component Analysis (PCA) (autoescaled data) using Chemostat V.2. Statistical analysis was performed after the verification of the homogeneity of variance and normality of residuals.

RESULTS AND DISCUSSION

The physicochemical parameters of the wines are presented in Table I. All parameters lie within the

identity and quality standards established by Brazilian legislation (MAPA, 2018). The parameters were presented in a similar way throughout all of the winemaking processes.

Table I

Physicochemical parameters of 'Marselan' red wines made by different winemaking processes

Parâmetros físico-químicos dos vinhos tintos 'Marselan' obtidos por diferentes processos de vinificação

Physicochemical parameters	TV	IV	CW	CWUS
Density	0.9954	0.9947	0.9958	0.9954
Alcoholic strength by volume (%)	12.6	12.0	12.6	12.6
Total acidity (g/L tartaric acid)	8.5	7.3	7.5	6.9
Volatile acidity (g/L acetic acid)	1.0	1.2	1.0	1.0
pH	3.5	3.4	3.6	3.5
Dry extract (g/L)	29.0	25.7	30.0	29.0
Reducing sugar (g/L)	3.9	3.2	3.5	3.4
Free sulfur dioxide (mg/L)	41.0	54.4	69.2	56.4
Total sulfur dioxide (mg/L)	129.2	132.8	124.0	143.6

TV – Thermovinification; IV – *Integrale* Vinification; CW – Classic winemaking; CWUS – Classic winemaking with ultrasound application.

Sixty-three volatile compounds were tentatively identified and semi-quantified. The volatile compounds belong to different chemical groups such as esters, ketones, acids, alcohols, terpenes, volatile phenols and sulfur compounds. The identified and semi-quantified compounds are shown in Table II. The main groups of volatile compounds found in this work were esters, alcohols and acids.

Esters develop from yeast metabolism during fermentation or naturally in grape berries (Perestrelo *et al.*, 2006; Vilanova *et al.*, 2013). The vinification process should have been the only factor to cause differences among the four wines since the grape and yeast used during fermentation remained constant throughout the study. Moreover, possible interactions between matrix and volatile compounds may increase or decrease flavor perception (Lorrain *et al.*, 2013). The majority compounds are ethyl acetate, isoamyl acetate, ethyl hexanoate, ethyl lactate, ethyl octanoate, ethyl decanoate and diethyl succinate. In general, these compounds showed higher concentrations in TV. Diethyl succinate and ethyl lactate were exceptions to this observation. Diethyl succinate and ethyl lactate were below the concentration threshold. For ethyl acetate and ethyl hexanoate, the concentrations were higher than the odor threshold. IV presented below the odor threshold for isoamyl acetate and ethyl decanoate, and presented above this value with TV (Peinado *et al.*,

2004; Fariña *et al.*, 2015). For minority esters, there was variation between the winemaking processes, which was better observed by chemometric analysis.

Alcohols are an important group of volatile compounds in wine. The main alcohols identified in our study were isobutyl alcohol, isopentyl alcohol, hexanol and phenylethyl alcohol, showing higher concentrations in IV and CWUS wines. The presence of C6 compounds, such as 1-hexanol and (E)3-hexen-1-ol, were observed. These compounds contribute to vegetal characteristics in wine (Gómez *et al.*, 1995; Vilanova *et al.*, 2013). (E)3-hexen-1-ol did not show significant differences between the winemaking processes and 1-hexanol, presented higher concentrations in CWUS and CW processes. Both did not reach the odor threshold (Guth, 1997).

Seven acids were identified. The majority of compounds were composed of acetic and octanoic acids, which did not exceed the odor threshold (Guth, 1997; Dominguez and Agosin, 2010). At low concentrations, acids improve aroma quality and complexity (Shinohara, 1985; Fariña *et al.*, 2015).

Statistical analysis was carried out to better distinguish the wines made by different winemaking processes due to the large number of variables present in this study. Considering that few variables hold the most important chemical information, the Principal Component Analysis (PCA) was used to provide fast

Table II

Volatile profile of 'Marselan' red wines detected by GC-FID and GC-MS with retention index, mean concentration and standard deviation

Perfil volátil dos vinhos tintos 'Marselan' detectados via GC-FID e GC-MS, contendo índice de retenção, concentração média e desvio padrão

Compound Name		TV (µg/L)	IV (µg/L)	CW (µg/L)	CWUS (µg/L)	KI ZB-WAX
K	Ketones					
K1	Acetone	9.7 ± 0.5 ^a	5.0 ± 0.4 ^b	8.1 ± 0.5 ^a	8.8 ± 1.3 ^a	809
K2	2,3-Butanedione	17.8 ± 1.2 ^a	17.6 ± 2.6 ^a	19.3 ± 1.4 ^a	31.5 ± 4.6 ^b	946
K3	3-Octanone	20.0 ± 0.5 ^a	21.4 ± 0.7 ^a	21.7 ± 0.3 ^a	20.1 ± 1.3 ^a	1245
K4	Cis-Geranylacetone	8.8 ± 1.1 ^a	9.1 ± 0.4 ^a	10.3 ± 0.6 ^a	9.8 ± 0.4 ^a	1869
E	Esters					
E1	Ethyl acetate	1115 ± 55.3 ^a	1193 ± 128.9 ^a	903.8 ± 29.4 ^b	860.9 ± 12.2 ^b	895
E2	Ethyl 2-methyl-propanoate	62.3 ± 3.6 ^a	38.2 ± 1.5 ^b	36.7 ± 3.4 ^{bc}	74.4 ± 8.7 ^a	936
E3	n-Propyl acetate	31.7 ± 2.3 ^a	19.3 ± 1.2 ^b	27.5 ± 1.1 ^{ab}	39.4 ± 7.7 ^a	942
E4	Isobutyl acetate	5.1 ± 0.5 ^{abc}	5.5 ± 0.6 ^b	3.9 ± 0.1 ^c	3.8 ± 0.3 ^{abc}	971
E5	Ethyl butanoate	2.3 ± 0.3 ^a	2.1 ± 0.2 ^a	1.4 ± 0.2 ^a	2.0 ± 0.3 ^a	1010
E6	Ethyl 2-methylbutanoate	4.0 ± 0.3 ^a	4.4 ± 0.4 ^a	2.1 ± 0.1 ^b	3.0 ± 0.4 ^{ab}	1029
E7	Isoamyl acetate	92.2 ± 7.3 ^a	10.3 ± 0.6 ^b	69.5 ± 1.3 ^c	57.8 ± 7.0 ^{cd}	1102
E8	Methyl hexanoate	1.5 ± 0.1 ^a	1.3 ± 0.1 ^a	0.8 ± 0.1 ^a	1.4 ± 0.1 ^a	1178
E9	Ethyl hexanoate	146.5 ± 14.9 ^a	128.8 ± 7.8 ^{ab}	111.6 ± 2.3 ^b	120.6 ± 11.8 ^{ab}	1230
E10	Hexyl acetate	6.3 ± 0.6 ^a	5.2 ± 0.1 ^a	3.0 ± 0.2 ^b	2.5 ± 0.2 ^{bc}	1266
E11	Ethyl heptanoate	2.0 ± 0.2 ^a	2.5 ± 0.3 ^a	2.0 ± 0.2 ^a	2.4 ± 0.2 ^a	1323
E12	Ethyl 2-hexenoate	9.0 ± 0.2 ^a	8.3 ± 0.5 ^a	1.8 ± 0.2 ^b	1.5 ± 0.1 ^{bc}	1330
E13	Ethyl lactate	112.8 ± 6.4 ^a	93.4 ± 8.7 ^b	136.1 ± 4.2 ^c	155.9 ± 3.5 ^d	1340
E14	Methyl octanoate	2.0 ± 0.1 ^a	1.7 ± 0.01 ^a	2.2 ± 0.2 ^a	2.9 ± 0.2 ^a	1380
E15	Ethyl octanoate	483.4 ± 70.3 ^a	365.0 ± 33.0 ^b	237.7 ± 13.9 ^c	284.8 ± 43.6 ^{bc}	1434
E16	Isoamylhexanoate	2.7 ± 0.1 ^a	3.8 ± 0.4 ^b	5.0 ± 0.3 ^c	4.3 ± 0.2 ^{bc}	1480
E17	Ethyl 3-hydroxybutanoate	2.8 ± 0.3 ^a	2.3 ± 0.01 ^a	2.3 ± 0.1 ^a	1.7 ± 0.1 ^a	1519
E18	Ethyl nonanoate	2.2 ± 0.1 ^a	2.0 ± 0.3 ^a	2.6 ± 0.1 ^a	2.5 ± 0.1 ^a	1527
E19	Ethyl 2-hydroxyhexanoate	35.1 ± 3.8 ^a	18.0 ± 2.4 ^b	32.6 ± 3.0 ^a	36.3 ± 3.0 ^a	1543
E20	Ethyl 3-(methylthio)propionate	9.6 ± 0.9 ^a	9.9 ± 0.01 ^a	10.7 ± 0.7 ^a	12.8 ± 1.3 ^b	1570
E21	Isoamyl lactate	3.5 ± 0.3 ^a	3.0 ± 0.4 ^{ab}	4.1 ± 0.5 ^{ac}	5.0 ± 0.4 ^c	1573
E22	Ethyl 2-furancarboxylate	11.5 ± 1.0 ^a	12.2 ± 0.6 ^a	8.7 ± 0.5 ^b	8.8 ± 1.0 ^b	1620
E23	Ethyl decanoate	206.5 ± 21.8 ^a	122.9 ± 14.2 ^b	56.9 ± 3.0 ^c	84.5 ± 5.8 ^c	1640
E24	Ethyl benzoate	16.9 ± 1.0 ^a	13.0 ± 1.6 ^{ab}	8.3 ± 0.2 ^{bc}	8.3 ± 0.5 ^{cd}	1664
E25	Diethyl succinate	226.2 ± 16.6 ^a	227.9 ± 15.3 ^a	489.2 ± 45.4 ^b	398.9 ± 5.9 ^c	1683
E26	Ethyl benzeneacetate	16.7 ± 1.0 ^a	21.1 ± 0.5 ^b	7.5 ± 0.3 ^c	7.2 ± 0.3 ^c	1786
E27	Ethyl myristate	4.1 ± 0.1 ^a	3.8 ± 0.6 ^{bc}	2.1 ± 0.1 ^b	3.7 ± 0.1 ^c	2063
E28	Ethyl pentadecanoate	2.3 ± 0.2 ^a	2.2 ± 0.1 ^a	2.2 ± 0.2 ^a	2.7 ± 0.01 ^a	2191
E29	Ethyl hexadecanoate	45.1 ± 5.0 ^a	45.5 ± 7.0 ^a	15.1 ± 2.1 ^b	21.8 ± 1.6 ^b	2285
E30	Ethyl hydrogen succinate	2.6 ± 0.2 ^a	3.2 ± 0.3 ^{ab}	2.0 ± 0.1 ^{ac}	1.8 ± 0.1 ^{ac}	2350
E31	Methyl stearate	14.2 ± 1.6 ^a	12.7 ± 1.7 ^a	24.9 ± 4.5 ^b	28.8 ± 3.9 ^b	2401

Table II
(continued)

Compound Name		TV (µg/L)	IV (µg/L)	CW (µg/L)	CWUS (µg/L)	KI ZB-WAX
H	Alcohols					
H1	Isobutyl alcohol	273.5 ± 12.6 ^{abc}	313.6 ± 24.0 ^b	251.5 ± 10.2 ^c	285.5 ± 6.6 ^{abc}	1077
H2	Butyl alcohol	8.4 ± 0.4 ^a	6.3 ± 0.6 ^b	11.0 ± 0.4 ^c	12.0 ± 0.4 ^c	1136
H3	Isopentyl alcohol	3154 ± 186.3 ^a	3500 ± 273.3 ^{ab}	2943 ± 132.4 ^a	3574 ± 74.4 ^{ab}	1216
H4	2-Heptanol	2.5 ± 0.1 ^a	2.0 ± 0.01 ^a	4.9 ± 0.2 ^b	5.9 ± 0.1 ^b	1318
H5	1-Hexanol	231.9 ± 13.0 ^a	220.4 ± 16.9 ^a	281.0 ± 12.1 ^b	313.2 ± 5.4 ^b	1351
H6	(E)3-hexen-1-ol	2.5 ± 0.1 ^a	2.6 ± 0.4 ^a	3.4 ± 0.2 ^a	4.2 ± 0.01 ^a	1360
H7	1-Octen-3-ol	33.4 ± 1.1 ^a	26.9 ± 3.1 ^b	34.4 ± 0.6 ^a	48.3 ± 2.5 ^c	1462
H8	2-Ethyl-hexyl alcohol	2.3 ± 0.1 ^a	1.7 ± 0.3 ^a	2.0 ± 0.1 ^a	7.0 ± 0.8 ^b	1496
H9	2-Nonanol	6.1 ± 0.4 ^a	5.5 ± 0.3 ^a	8.4 ± 0.3 ^b	11.9 ± 0.9 ^c	1521
H10	1-Octanol	2.9 ± 0.2 ^a	2.3 ± 0.01 ^a	3.1 ± 0.3 ^a	4.0 ± 0.2 ^b	1567
H11	(E)2-Octen-1-ol	15.4 ± 1.4 ^a	11.9 ± 1.4 ^a	29.0 ± 2.2 ^b	35.1 ± 1.8 ^c	1618
H12	1-Decanol	7.2 ± 0.2 ^a	7.2 ± 0.7 ^a	6.9 ± 0.1 ^a	8.1 ± 0.8 ^a	1755
H13	Benzyl Alcohol	8.9 ± 1.0 ^a	7.6 ± 1.3 ^{ab}	13.2 ± 1.4 ^c	12.7 ± 0.9 ^{ac}	1882
H14	Phenylethyl alcohol	1320 ± 114.7 ^a	1470 ± 78.4 ^a	744.3 ± 100.4 ^b	1044 ± 15.9 ^c	1919
T	Terpenes					
T1	Linalool	4.6 ± 0.5 ^a	4.7 ± 0.6 ^{ab}	4.1 ± 0.4 ^a	3.5 ± 0.3 ^{ac}	1549
T2	(-)-4-Terpineol	7.1 ± 0.9 ^a	4.3 ± 0.7 ^b	11.6 ± 0.5 ^c	12.0 ± 1.3 ^c	1594
T3	(-)-Myrtenol	3.3 ± 0.2 ^a	3.4 ± 0.3 ^a	3.1 ± 0.2 ^{ab}	2.7 ± 0.2 ^b	1797
A	Acids					
A1	Acetic acid	105.0 ± 4.8 ^a	114.7 ± 13.3 ^{ab}	91.1 ± 3.1 ^{ac}	92.8 ± 4.7 ^{ac}	1459
A2	Butanoic acid	1.9 ± 0.2 ^a	2.0 ± 0.1 ^a	1.9 ± 0.01 ^a	2.4 ± 0.2 ^a	1624
A3	Hexanoic acid	72.2 ± 6.5 ^a	72.5 ± 4.2 ^a	47.3 ± 5.1 ^b	60.1 ± 1.9 ^{ac}	1858
A4	Ethylhexanoic acid	2.4 ± 0.1 ^a	1.8 ± 0.1 ^b	3.1 ± 0.1 ^a	2.3 ± 0.1 ^a	1975
A5	Octanoic acid	179.1 ± 15.0 ^a	181.8 ± 10.2 ^a	72.9 ± 9.2 ^b	94.7 ± 3.6 ^b	2074
A6	Tetradecanoic acid	2.1 ± 0.2 ^a	1.7 ± 0.01 ^a	1.8 ± 0.2 ^a	1.9 ± 0.1 ^a	2710
A7	n-Hexadecanoic acid	3.2 ± 0.4 ^a	4.8 ± 0.7 ^{ab}	5.4 ± 0.6 ^b	4.4 ± 0.5 ^{ab}	2855
O	Others					
O1	Furfural	6.8 ± 0.1 ^a	6.2 ± 0.2 ^a	7.9 ± 0.2 ^b	7.4 ± 0.6 ^b	1490
O2	Benzaldehyde	38.6 ± 2.5 ^a	28.3 ± 0.2 ^a	104.2 ± 9.9 ^b	140.0 ± 5.0 ^c	1508
O3	Methionol	11.3 ± 0.5 ^a	11.5 ± 1.6 ^a	8.7 ± 0.9 ^b	9.4 ± 0.7 ^{ab}	1718
O4	4-Ethylphenol	3.0 ± 0.2 ^a	3.0 ± 0.4 ^a	1.7 ± 0.2 ^b	2.5 ± 0.2 ^{ab}	2206

Results expressed as concentration (mean ± standard deviation); TV – Thermovinification; IV – *Integrale* Vinification; CW – Classic winemaking; CWUS – Classic winemaking with ultrasound application; KI ZB-WAX - Experimental linear retention index on capillary column ZB-Wax; abcd - Different letters in the same row indicate significant differences (p<0.05; Tukeys's test).

and efficient information about the similarities and differences between the samples (Morgano *et al.*, 1999).

PCA was used to better examine the compounds which underwent changes in their concentration due to the different winemaking processes applied. The PCA included only the compounds that show statistical differences (Figure 1). Of the sixty-three

volatile compounds, only thirteen did not show any significant differences. PC1 and PC2 explained 88.52% of the total variance. PC1 separated CW and CWUS wines from TV and IV. PC2 separated CW and IV wines from TV and CWUS. This division was expected since CW and CWUS were elaborated in similar ways. However, the temperature applied to TV and the ultrasound applied to CWUS resulted in similar levels of extraction.

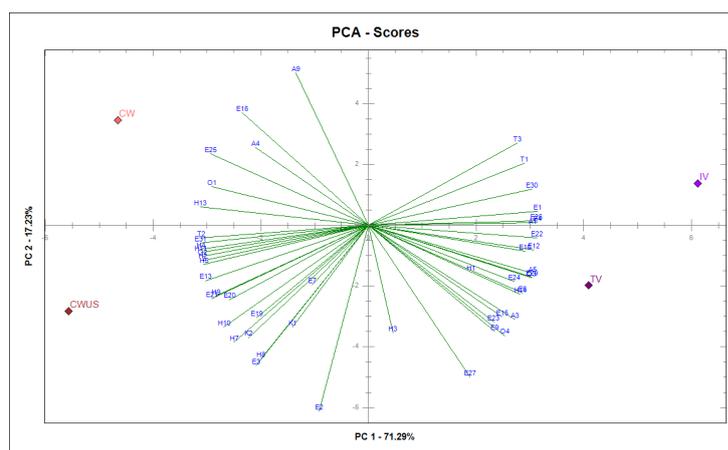


Figure 1. PCA plot of volatile compounds of ‘Marselan’ red wines made by different winemaking processes.

Representação da PCA dos compostos voláteis dos vinhos tintos ‘Marselan’ resultantes de diferentes processos de vinificação.

TV: thermovinification; IV: *integrale* vinification; CW: classic winemaking; CWUS: classic winemaking with ultrasound application. The legend for the volatile compounds codes can be seen in Table II.

Greater influence of ethyl esters and acetates was observed for TV. This finding coincides with the study carried out by Geffroy *et al.* (2015), who verified an increase in the concentration of these compounds; using similar methodology in different grape varieties. This result is expected due to applied heat during thermovinification, which causes the softening of berry peels and generates a higher probability for aromatic precursors to break. Consequently, this enables the release of more organic acids, ethyl esters and acetates contributing to a sweet and fruity aroma. Different esters, even at low concentrations, can cause changes in the aroma of wines (Pineau, 2007) supporting the slight variations found in some compounds in this work. Compounds such as isobutyl acetate and ethyl-3-hydroxybutanoate were identified as an indirect influence on the aroma of wines, even at low concentrations (Lytra *et al.*, 2013). Throughout this research, these compounds presented higher concentrations in the TV and CWUS wines, further explaining their strong fruity characteristics.

CWUS presented greater influence on alcohols. This result is consistent with the study performed by Plaza *et al.* (2019). The application of ultrasound causes changes such as extraction of bioactive compounds in a shorter time. It also affects the volatile composition as it causes the rupture of cells and increases the rate of mass transfer of several compounds, including aroma precursors (Plaza *et al.*, 2019).

To observe the impact of volatile compounds on wines, a quantitative descriptive sensory analysis was performed.

All of the winemaking processes resulted in a similar aroma profile among the wines (Figure 2), but some differences could be distinguished. TV and CWUS had a more similar aroma profile, with a greater perception of a fruity aroma, overmaturation aroma and aromatic intensity and complexity. Undesirable and vegetal aromas were not perceived as strongly. These perceptions were inverted for CW and IV. Significant differences were observed for vegetal and undesirable aromas. In order to obtain a better relationship between the sensory aspect and the

different winemaking processes, a chemometric approach was used; Principal Component Analysis (PCA).

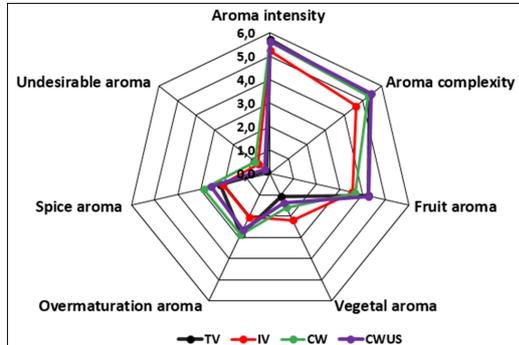


Figure 2. Sensorial profile of 'Marselan' red wines made by different winemaking processes.

Perfil sensorial dos vinhos tintos 'Marselan' obtidos por diferentes processos de vinificação.

The PCA biplot for the Quantitative Descriptive Analysis (Figure 3) revealed the following information; PC1 showed 70.05% of the total variance and PC2 showed 26.51%, totaling 96.56%.

PC1 separated wines from TV, CWUS, and CW from IV, while PC2 separated wines from TV, CWUS, and IV from CW, demonstrating some differences

between the three suggested winemaking processes and CW. This division confirms the previous explanation of a greater influence from the fruity aroma on TV wines, aroma intensity and complexity and overmaturation aroma in CWUS wines, and vegetal and undesirable aromas in IV and CW wines. This division and the aromatic descriptors cited by the judges in Table III further explain the aromatic compounds found in the wines. It is important to keep in mind that the intensity of each compound depends on its concentration threshold and set of aromatic compounds.

CWUS showed similar scores to TV in sensory analysis in terms of intensity, complexity and fruity aroma profiles. Similar behavior can be explained by the ultrasound pretreatment, which causes cellular rupture and increase of mass transfer rates (Mason, 1998; Toma *et al.*, 2001; Ferraretto and Celotti, 2016). This phenomenon can be compared with the temperature application in TV. CWUS pretreatment was more abrupt than TV, because it caused additional extraction of several other compounds, resulting in greater distinction of aromas in sensory analysis as shown in Table III. Fruity aromas and other compounds, such as ethereal conferred by alcohols, butter (2,3-butanedione) and almond (benzaldehyde) tones, give greater aromatic complexity (Zea *et al.*, 2001; Fariña *et al.*, 2015; Lyu *et al.*, 2019).

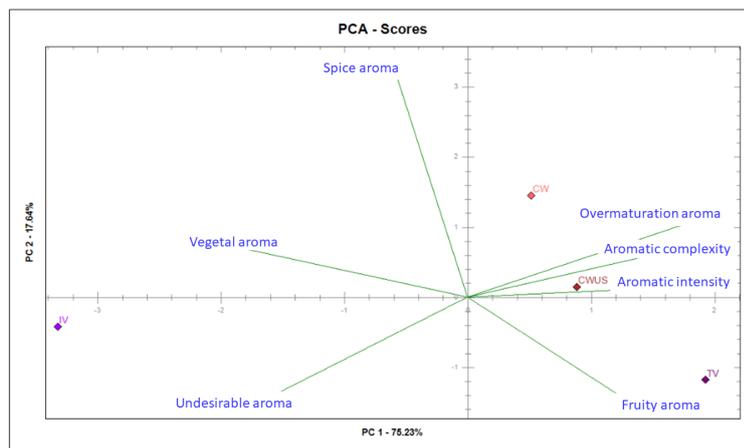


Figure 3. PCA plot of sensory analysis of 'Marselan' red wines made by different winemaking processes.

Gráfico de PCA da análise sensorial dos vinhos tintos 'Marselan' obtidos por diferentes processos de vinificação.

◆TV: thermovinification; ◆IV: integrale vinification; ◆CW: classic winemaking; ◆CWUS: classic winemaking with ultrasound application.

Table III

Aromatic descriptors found in the quantitative descriptive analysis cited by the judges and its prevalence

Descritores aromáticos encontrados na análise descritiva quantitativa pelos provadores e sua prevalência

Vinification Technique	Fruit Aroma (%)	Vegetal Aroma (%)	Overmaturation Aroma (%)	Spice Aroma (%)	Undesirable Aroma (%)
Thermovinification (TV)	Plum - 42.8 Strawberry - 16.9 Red fruits - 9.5 Raspberry - 7.1 Blackberry - 9.5 Cassis - 4.7 Neutral - 4.7 Blueberry - 2.4 Guava - 2.4	Not detected - 38 String bean - 16.7 Bell pepper - 14.3 Asparagus - 7.1 Twig - 4.7 Broccoli - 2.4 Cabbage - 2.4 Mint - 2.4 Hay - 2.4 Herbs - 2.4 Eucalyptus - 2.4 Pencil - 2.4 Withe - 2.4	Raisin - 33.3 Neutral - 21.4 Compote - 16.7 Jelly - 9.5 Honey - 9.5 Liqueur cassis - 4.8 Vinous - 4.8	Neutral - 35.6 Pepper - 26.2 Cinnamon - 12.0 Clove - 7.1 Nutmeg - 7.1 Herbs - 4.8 Mushroom - 4.8 Vanilla - 2.4	Not detected - 95.2 Oxidized - 2.4 Herbaceous - 2.4
Integrale Vinification (IV)	Plum - 45 Neutral - 12.5 Red fruits - 10 Blackberry - 7.5 Strawberry - 7.5 Cherry - 7.5 Fig - 2.5 Tropical fruit - 2.5 Guava - 2.5 Jaboticaba - 2.5	Bell pepper - 29.4 Not detected - 24.4 String bean - 21.9 Asparagus - 4.9 Dry straw - 4.9 Twig - 4.9 Withe - 2.4 Herbaceous - 2.4 Grass - 2.4 Coconut - 2.4	Not detected - 35.7 Raisin - 33.3 Compote - 11.9 Jelly - 9.5 Liqueur of red fruits - 4.8 Cassis - 2.4 Honey - 2.4	Not detected - 26.8 Pepper - 14.9 Mint - 12.1 Clove - 9.7 Cinnamon - 7.3 Mushroom - 7.3 Nutmeg - 7.3 Fine herbs - 4.9 Honey - 4.9 Vanilla - 2.4 Chocolate - 2.4	Not detected - 80.8 Herbaceous - 12.0 Twig - 2.4 Oxidized - 4.8
Classic winemaking (CW)	Plum - 42.8 Not detected - 14.3 Blackberry - 11.9 Red fruits - 9.5 Strawberry - 7.14 Currant - 4.8 Physalis - 2.4 Tropical fruit - 2.4 Cherry - 2.4 Raspberry - 2.4	Not detected - 40.3 Bell pepper - 21.4 String bean - 9.5 Dry straw - 4.8 Withe - 4.8 Broccoli - 4.8 Asparagus - 4.8 Cabbage - 2.4 Pepper - 2.4 Twig - 2.4 Mint - 2.4	Not detected - 33.4 Raisin - 30.9 Jelly - 9.5 Burnt coconut - 9.5 Honey - 7.1 Liqueur - 4.8 Grape jelly - 2.4 Marmalade - 2.4	Pepper - 30.9 Clove - 19.1 Not detected - 14.3 Cinnamon - 9.5 Herbs - 9.5 Mint - 7.1 Mushroom - 4.8 Nutmeg - 2.4 Burnt coconut - 2.4	Not detected - 76.1 Herbaceous - 14.3 Resin - 2.4 Reduced wine - 2.4 Burnt wine - 2.4 Twig - 2.4
Classic winemaking with ultrasound application (CWUS)	Plum - 40.5 Blackberry - 21.4 Strawberry - 14.3 Raspberry - 7.0 Red fruits - 4.8 Not detected - 4.8 Cherry - 2.4 Currant - 2.4 Cassis - 2.4	Not detected - 45.1 Asparagus - 14.3 String bean - 12 Bell pepper - 9.5 Twig - 7.1 Dry straw - 4.8 Grass - 2.4 Pepper - 2.4 Coriander - 2.4	Raisin - 35.6 Not detected - 26.2 Jelly - 9.5 Honey - 7.1 Compote - 7.1 Liqueur - 4.8 Marmalade - 4.9 Cassis - 2.4 Grape jelly - 2.4	Pepper - 26.0 Not detected - 19.0 Cinnamon - 14.3 Clove - 12 Nutmeg - 7.1 Mint - 4.8 Vanilla - 4.8 Herbs - 4.8 Prune - 2.4 Leather - 2.4 Mushroom - 2.4	Not detected - 85.8 Herbaceous - 12.0 Resin - 2.2

Undesirable and vegetal aromas had a greater influence on IV and CW due to the higher perception of sulfurous and green aromas. This result proves that small differences in the concentration of certain compounds can greatly influence the final aromas of wine, since these two wines had less influence from esters.

It is important to highlight that IV wine should have given the perception of a spice character since it was the only wine to undergo contact with an oak barrel. The absence of the spice aroma could be linked to the brief length of time the wine was in contact with wood. The aroma profile of IV wine may be a result of different extraction rates, oxidation and reduction phenomena, caused by the use of oak barrels, like the reduction of the volatility of certain compounds, such as esters, alcohol and aldehydes, due the wood contact and the lack of formation of others compounds which give pleasant aromas such as lactones, vanillin, among others (Conner *et al.*, 1999; Escalona *et al.*, 1999, 2002; Gagliole, 2013). The aroma of this wine can be improved by increasing maturation time in oak barrels after the stabilization period. It is important to highlight, this technique is being used in many wineries, but no scientific studies have been found on the subject.

It is important to emphasize 'Marselan' wines may have a more vegetal aroma due to its crossing between 'Grenache' and 'Cabernet Sauvignon'. This can be attributed to the green tones found in the wines, mainly for IV. In addition to the influence of the grape variety, cold regions also influence the vegetal aroma in the wines due to slower grape ripening (Miele and Rizzon, 2011). To decrease the perception of the vegetal aroma, it is necessary to use processes which promote the extraction of compounds and give more fruity tones such as ethyl esters and acetates.

Observations showed the winemaking process influenced the concentration of the compounds, causing changes in the sensory characteristics of wines. The different methods used did not allow the detection of different compounds between wines, only changes in their concentrations. This is expected since all other variables were controlled, including grape variety and crop, which are considered the major causes of change in the volatile composition of wines (Mihnea *et al.*, 2014; De Castilhos *et al.*, 2019; Ma *et al.*, 2020; Piras *et al.*, 2020). However, the concentration of volatile compounds and the physicochemical characteristics caused great differences between the wines, demonstrating the effect of the winemaking processes on the wine quality.

CONCLUSIONS

Chemometric analysis was useful for the study and showed a clear distinction between the winemaking processes. It was possible to verify the impact of different winemaking processes on the volatile composition and sensory characteristics of the wines. The wines from TV and CWUS presented compounds with fruity and overmaturation tones due to the temperature applied in TV wines. This caused the softening of berry peels and generated a higher probability of aromatic precursors to break. The ultrasound pretreatment increased the mass transfer rate. In order to obtain better results for IV, it is necessary for the wine to be in contact with the oak barrels for a longer period. This prolonged contact will help mask the strong vegetal aroma and increase the extraction of compounds responsible for the production of spicy aromatic notes. The TV and CWUS winemaking processes promote fruity, spicy, and overmaturation notes and can help to improve the vegetal aroma from the 'Marselan' cultivar. This study is important because the consumption of 'Marselan' wine is increasing, especially in new world countries. Winemaking processes directly affect the aromatic quality of wines due to different extraction rates of several compounds, contributing to various aromatic profiles. This study contributes to improving the quality of elaborated wines, allowing winemakers to choose new winemaking processes to enhance the aromatic compounds naturally present in the grapes.

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