



Article

Effect of Thermovinification Temperature on Phenolic Compounds and Colour of Syrah Wine

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Abstract: Background: Thermovinification is a non-conventional winemaking practice that replaces the traditional method of grape maceration. Methods: This study evaluated the influence of thermovinification temperature on the quality of Syrah wines. The treatments included traditional winemaking with 7 days of maceration during alcoholic fermentation at 23 °C (TW-control); and thermovinification for 2 h at 55 °C (TV55), 65 °C (TV65), and 75 °C (TV75). The red wines were made through microvinification (10-litre glass). Phenolic compounds (n = 26) were quantified by high-performance liquid chromatography and a colour analysis using the CIELab/CIEL*C*h systems and a sensory analysis was conducted to evaluate the acceptability of the thermovinified wine. Results: The results indicate that thermovinification increased the content of bioactive compounds and intensified the colour of the wine, reducing L* and a*. However, the content of phenolic acids decreased, except for trans-caftaric acid, which was approximately 50 times higher. A higher temperature of thermovinification (75 °C) promoted the degradation of all anthocyanins. Among flavonols, kaempferol-3-O-glucoside, quercetin-3-β-D-glucoside, and isorhamnetin-3-O-glucoside were higher in TV65 and TV75 wines. Greater amounts of stilbenes were quantified in TV65. Among the flavan-3-ols, TV75 stood out, especially for (+)-catechin, (-)-epicatechin, procyanidin A2, and procyanidin B1. Conclusions: The thermovinification at 65 °C is optimal for minimising anthocyanin degradation and improving Syrah wine quality.

Keywords: thermomaceration; bioactive compounds; tropical red wines; winemaking practises



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1. Introduction

Polyphenols are essential compounds in red wines, as they are involved in their sensory properties (colour, flavour, body, astringency, and bitterness) [1], ageing behaviour, and the health benefits attributed to moderate wine consumption [2].

Several factors affect the content and composition of phenolic compounds in wines, such as grape variety, the profile of phenolic compounds in the cultivar as a function of the environmental conditions of the vineyard, and the choice of the harvest date [3]. Grapes play a vital role in the chemical profile of red wine [4]. However, one of the most significant factors is the winemaking process itself [3]. The duration and temperature of the maceration process have major effects on the extraction of phenolic compounds from grapes [5]. Thus,

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maceration techniques, combined with initial grape quality, determine the amount and stability of phenolic compounds such as tannins and anthocyanins, which influence the composition and sensory quality of red wines [6].

During traditional winemaking, only approximately 40% of the anthocyanins and 20% of the tannins from grape skins are transferred to red wine [7,8]. This limited extraction is primarily due to the insufficient permeability of the cell walls and cytoplasmic membranes [9,10]. Numerous studies have focused on alternative practises capable of weakening cell barriers and increasing the polyphenol content in red wines [11,12], such as thermovinification.

The first work on heat treatment of musts was conducted more than 60 years ago in California, both in a laboratory and artisanally [13]. The development of industrial heating systems in the 1970s and the large number of associated research papers published in the same decade [14–17] reflect the wine industry's strong interest in this technology.

Thermovinification can be used for red wines to increase the content of certain phenolic compounds and inhibit the action of oxidising enzymes such as laccase and polyphenol oxidase [18]. Additionally, heating reduces the activity of pectolytic enzymes responsible for methanol production [19]. The thermovinification technique also reduces the alcoholic fermentation and maceration process time, facilitating the migration of grape metabolites to the must (sugars, organic acids, amino acids, and anthocyanins) [20].

The heat applied during thermovinification destroys the cell membranes of the grape skin, releasing pigments, tannins, and other phenolic compounds. Therefore, the extraction yield depends on the temperature, generally within the range of 65 °C for 2 h [21]. This technique is becoming increasingly popular to produce red wines. The volume of wine produced in France alone through thermovinification is estimated to exceed 750 million litres [20]. For red wines intended to be consumed young, and when colour intensity is a concern, thermovinification can help enhance the colour intensity and shelf life of the beverage [22,23].

The enhanced extraction of phenolic compounds during winemaking offers opportunities to improve wine quality and nutraceutical value, aligning with the growing interest of consumers in high-quality red wines with potential health benefits [24]. Thermovinification has emerged as an alternative technique for producing wines with better structure, colour stability, and nutraceutical properties, while simultaneously optimising the winemaking process. To improve the effectiveness of thermovinification, we evaluated the influence of different thermovinification temperatures on the phenolic profile and colour of Syrah wine.

2. Materials and Methods

2.1. Raw Material

A total of 142 kg of Syrah grapes grown in the experimental area of Embrapa (09 $^{\circ}$ 09′ S, 40 $^{\circ}$ 22′ W, 365.5 m, Petrolina, PE, Brazil) was used. The grapes were harvested in March 2019, with average values of soluble solids content of 21.6 $^{\circ}$ Brix, a pH of 3.5, and titratable acidity of 6.4 g/L (expressed as tartaric acid). The climate of the region is classified as BSwh according to Köppen, corresponding to a very hot semi-arid region [25], with an average annual air temperature of 26 $^{\circ}$ C, relative humidity of 64%, and annual rainfall of 549 mm. Vines were grown in a vertical shoot positioning trellis system, grafted onto Paulsen 1103 rootstocks, and irrigated using drip irrigation.

2.2. Winemaking Process

The red wines were made through microvinification in 10-litre glass carboys with glass air-lock valves. The Syrah grapes were first homogenised and equally divided into each of the following treatments, as illustrated in Figure 1: thermovinification at 55 $^{\circ}$ C (TV55), thermovinification at 65 $^{\circ}$ C (TV65), thermovinification at 75 $^{\circ}$ C (TV75), and traditional winemaking (TW—control). All microvinifications were conducted in triplicate.

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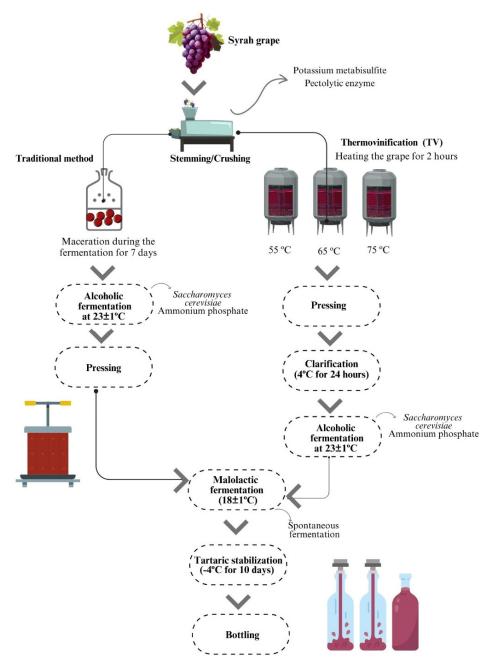


Figure 1. Flowchart for production of Syrah red wines using traditional vinification and thermovinification.

For the TW treatment, the grapes were destemmed, crushed, and subjected to simultaneous maceration (7 days) and alcoholic fermentation at 23 ± 1 °C. For the grapes in the other treatments (TV55, TV65, and TV75), after destemming and crushing, the grapes were transferred to a 50-litre stainless steel vat (West equipment, Juiz de Fora, MG Brazil) to be heated according to the temperature of each treatment, applying thermovinification for a period of 2 h. After heating, the musts were cooled and pressed using a vertical hydraulic press. Alcoholic fermentation was then started (23 °C) without the grape skins and seeds and after must clarification at a temperature of 4 °C for 24 h [21].

During the destemming process, potassium metabisulfite as a preservative (Amazon Group Ltd., Bento Gonçalves, RS, Brazil, 100 mg/Kg) and a pectolytic enzyme (Pectozim Rouge Gr®, Ever Brasil, Garibaldi, RS, Brazil, 0.02 mg/L) were added. For alcoholic fermentation, 0.20 g/L of the commercial yeast *Saccharomyces cerevisiae* (Maurivin PDM®, Mauri Yeast Pty Ltd., Camellia, NSW, Australia) and 0.20 g/L of ammonium phosphate as a nutrient (Gesferm®, Amazon Group Ltd., Bento Gonçalves, RS, Brazil) were used.

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Following the completion of alcoholic fermentation, the wines underwent spontaneous malolactic fermentation at 18 \pm 1 °C for a month. Tartaric stabilisation was achieved through a cold treatment at -4 °C for 10 days. The bottling process was then carried out after free SO₂ was adjusted to 50 mg/L.

2.3. Phenolic Compound Profile

Twenty-six phenolic compounds were quantified in the samples in a single 60 min run using high-performance liquid chromatography (HPLC) (Waters Alliance e2695 model, Milford, CT, USA) coupled with a diode array (DAD) (280, 320, 360, and 520 nm) and fluorescence (280 nm excitation and 360 nm emission) detectors. The validated methods under the same analytical conditions by Natividade et al. [26] and Da Costa et al. [27] were used for the sample analyses. The separation of compounds was realised using a column and pre-column Gemini-NX C18 (150 mm \times 4.60 mm \times 3 μ m) and Gemini-NX C18 (4.0 mm \times 3.0 mm) (Phenomenex, Torrance, CA, USA), respectively. The gradient elution was applied with the mobile phases constituting 0.85% orthophosphoric acid (Fluka, Geneva, Switzerland) in ultrapure water (Purelab Option Q Elga System, Oxford, UK) and acetonitrile (J. T. Baker, Phillipsburg, NJ, USA, HPLC grade). The oven temperature was maintained at 40 °C and the flow rate was 0.5 mLmin $^{-1}$. Ten microliters of each wine was injected into the equipment after filtering through a 13 mm diameter nylon membrane with a pore size of 0.45 μ m.

The standards of (–)-epicatechin gallate; (–)-epigallocatechin gallate; (+)-catechin; (–)-epicatechin; procyanidins A2, B1, and B2; kaempferol-3-O-glucoside; quercetin-3- β -D-glucoside; isorhamnetin-3-O-glucoside; myricetin; rutin; malvidin-3-O-glucoside; peonidin-3-O-glucoside; delfinidin-3-O-glucoside; pelargonidin-3-O-glucoside; petunidin-3-O-glucoside; cyanidin-3-O-glucoside; and *trans*-resveratrol were acquired from Extrasynthese (Genay, France). Caffeic, *trans*-caftaric, ρ -coumaric, chlorogenic, and gallic acids; ε -viniferin; and piceatannol were purchased from Sigma-Aldrich (St. Louis, MO, USA); a ferulic acid standard was obtained from ChemService (West Chester, PA, USA) and *cis*-resveratrol from Cayman Chemical (Ann Arbor, MI, USA).

The total phenolic content was determined by the method of Singleton and Rossi [28] using a Folin–Ciocalteu reagent (Sigma-Aldrich, St. Louis MO, USA). The absorbance of the samples was read in a spectrophotometer (Thermo Fisher Scientific, Multiskan Go, Waltham, MA, USA) at 760 nm, and gallic acid (Vetec, RJ, Brazil) was used to obtain the calibration curve.

2.4. Colorimetric Parameters

The transmittance mode, illuminant D65, and a 10° angle were used to conduct the analyses. A portable colorimeter (Delta Color, São Leopoldo, RS, Brazil) and CIELab and CIEL*C*h systems [29] were applied to analyse the following parameters: the luminosity (L*), red/green component (a*), blue/yellow component (b*), saturation (C*), and hue angle (h).

2.5. Sensory Analysis—Consumer Test

The acceptability of red wines was evaluated by consumers recruited at IFSertão Pernambucano, Petrolina, PE, Brazil. After approval from the Research Ethics Committee (CAAE 67164022.2.0000.8052), in accordance with Resolution 466/12 of the National Health Council, Brazil, sixty regular wine consumers were selected, consisting of 55% women and 45% men, aged between 21 and 50 years. All subjects were informed about the research objectives and signed consent for their participation.

Samples of thirty millilitres (30 mL) were evaluated in ISO glasses coded with three-digit numbers. Sample assessments were conducted at $22 \pm 1\,^{\circ}\text{C}$ in individual booths under incandescent white lighting for acceptance and purchase intention tests. In the acceptance test, consumers evaluated the appearance, aroma, flavour, and overall impression of the wines. The 9-point hybrid hedonic scale proposed by Villanueva and Da Silva [30] and

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adjusted by Biasoto et al. [31] was used, with terms ranging from "1: extremely disliked" to "9: extremely liked." Purchase intention was assessed according to Meilgaard et al. [32] using a 5-point categorical scale (1: definitely would not buy, 2: probably would not buy, 3: uncertain about buying, 4: probably would buy, and 5: definitely would buy).

2.6. Statistical Analysis

Three bottles were analysed for each batch by treatment in triplicate. Data were subjected to an analysis of variance (ANOVA) and Tukey's test ($p \le 0.05$) using XLSTAT software version 2015 (Addinsoft Inc., Anglesey, UK, 2015). Colour parameter graphs were created using SPSS version 20.0 for Windows (SPSS, Chicago, IL, USA). For the total phenolics graph, OriginLab (Version 2010, Northampton, MA, USA) was employed. A principal component analysis (PCA) graph was prepared using XLSTAT software (Addinsoft Inc., Anglesey, UK, 2015).

3. Results and Discussion

3.1. Phenolic Compounds

Figure 2 shows the total phenolic compounds determined by spectrophotometry, expressed in mg/L GAE. The results showed that thermovinification increased the total phenolic content of the wine, with the most notable concentrations observed in the wines whose musts were subjected to temperatures of 65 °C and 75 °C (TV65 and TV75) at 3718.03 and 3916.24 mg/L GAE, respectively. The lowest concentration was found in winemaking with the traditional maceration practice (TW), with a concentration of 3311.27 mg/L GAE. In the study carried out by Atanackovic et al. [33] using thermovinification (60 °C/1 h and 80 °C/3 min), for all varieties analysed (Merlot, Cabernet Sauvignon, Pinot Noir, and Prokupac), the wines from the control treatment (maceration concomitant with fermentation for 14 days at 25 °C) presented the lowest concentrations of total phenolics (564.43–911.55 mg/L GAE). Except for wines from the Pinot Noir variety, the condition that best promoted the extraction of phenolic compounds was the use of thermovinification at 60 °C for 1 h—Merlot (1208.63 mg/L GAE), Cabernet Sauvignon (1410.97 mg/L GAE), and Prokupac (1159.37 mg/L GAE). Pinot Noir had its highest level of total phenolic concentration at 80 °C for 3 min (1196.66 mg/L GAE).

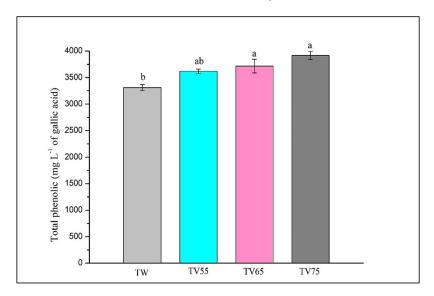


Figure 2. Total phenolic content measured using the Folin–Ciocalteu reducing capacity expressed in mg/L of gallic acid. Samples: TW, Syrah wine winemaking using traditional maceration; TV55, Syrah wine winemaking using thermovinification at 55 °C for 2 h; TV65, Syrah wine winemaking using thermovinification at 65 °C for 2 h; and TV75, Syrah wine winemaking using thermovinification at 75 °C for 2 h. Different letters indicate a significant difference between samples according to Tukey's test ($p \le 0.05$).

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Table 1 shows the phenolic compounds (n = 26) identified in the wines using HPLC-DAD. In general, compared with other maceration practises [24,34,35], the conditions of thermovinification applied in this study (55, 65, and 75 °C for 2 h) proved to be efficient in extracting phenolic compounds from the Syrah grapes.

Table 1. Phenolic profile of Syrah wines vinified using different thermovinification temperatures and the traditional method (conventional maceration).

Phenolic Compounds (mg/L) ^a		Treatments ^b		
	TW	TV55	TV65	TV75
Anthocyanins				
Pelargonidin-3-O-glucoside	2.89± 0.07 b	3.46 ± 0.49 a	$2.37 \pm 0.08 \text{ c}$	$1.48 \pm 0.10 \text{ d}$
Delfinidin-3-O-glucoside	$1.20 \pm 0.03 \mathrm{b}$	1.65 ± 0.27 a	$1.16 \pm 0.05 \mathrm{b}$	$0.80 \pm 0.04 c$
Malvidin-3-O-glucoside	30.61 ± 0.50 a	29.43 ± 4.39 a	$20.79 \pm 1.52 \mathrm{b}$	$11.68 \pm 0.77 \mathrm{c}$
Peonidin-3-O-glucoside	$1.93\pm0.42~\mathrm{ab}$	2.26 ± 0.59 a	$1.71 \pm 0.18 \mathrm{b}$	$0.95 \pm 0.18 \mathrm{c}$
Petunidin-3-O-glucoside	1.24 ± 0.06 a	$1.02 \pm 0.10 \mathrm{b}$	$0.96 \pm 0.14 \mathrm{b}$	$0.70 \pm 0.05 \mathrm{c}$
Cyanidin-3-O-glucoside	ND	ND	ND	ND
\sum Anthocyanins	37.87 mg/L	37.82 mg/L	26.99 mg/L	15.61 mg/L
Phenolic acids				
Gallic acid	28.26 ± 0.20 a	$24.46 \pm 5.27~{ m ab}$	$22.23 \pm 1.22 \mathrm{b}$	24.93 ± 0.17 ab
Caffeic acid	13.96 ± 0.35 a	$4.57 \pm 0.26 \ \mathrm{c}$	$5.15 \pm 0.22 \text{ c}$	$8.27 \pm 2.80 \mathrm{b}$
trans-caftaric acid	$2.64 \pm 0.19 \mathrm{b}$	132.17 ± 11.31 a	126.37 ± 27.3 a	119.07 ± 26.08
Chlorogenic acid	0.73 ± 0.01 a	ND	ND	ND
ρ-Coumaric acid	5.58 ± 0.27 a	$2.97 \pm 0.04 \mathrm{b}$	$1.40 \pm 0.09 c$	$2.33 \pm 1.14 \ bc$
Ferulic acid	$2.02\pm0.02~a$	$0.69\pm0.01~\mathrm{c}$	$0.72 \pm 0.02 \mathrm{b}$	$0.70 \pm 0.03 \ \mathrm{bc}$
\sum Phenolic acids	53.19 mg/L	164.86 mg/L	155.87 mg/L	155.30 mg/L
Stilbenes				
<i>cis</i> -resveratrol	$0.16 \pm 0.00 \text{ c}$	$0.17 \pm 0.01 \mathrm{bc}$	0.18 ± 0.10 a	$0.17 \pm 0.01~{ m ab}$
Piceatannol	$1.18 \pm 0.01 c$	$1.94 \pm 0.18 \mathrm{b}$	$2.28 \pm 0.04 a$	$2.25 \pm 0.04 a$
trans-resveratrol	$0.54\pm0.01~\mathrm{c}$	$0.59 \pm 0.06 \ ab$	0.62 ± 0.03 a	$0.57 \pm 0.03 \ \mathrm{bc}$
$arepsilon ext{-viniferin}$	ND	ND	ND	ND
∑ Stilbenes	1.88 mg/L	2.70 mg/L	3.08 mg/L	2.99 mg/L
Flavan-3-ols				
(–)-Epicatechin gallate	2.07 ± 0.19 a	$1.38 \pm 0.10 \text{ c}$	$1.37 \pm 0.08 \text{ c}$	$1.66 \pm 0.17 \mathrm{b}$
(–)-Epigalatocatechin gallate	15.79 ± 0.12 a	$13.32 \pm 2.76 \mathrm{b}$	$8.51 \pm 0.74 \mathrm{c}$	$6.00 \pm 0.32 \mathrm{d}$
(+)-Catechin	$17.82 \pm 1.06 \mathrm{c}$	$19.13 \pm 5.30 \text{ c}$	$29.94 \pm 3.60 \mathrm{b}$	35.01 ± 1.54 a
(−)-Epicatechin	$11.08 \pm 0.66 \mathrm{b}$	$11.25 \pm 3.31 \mathrm{b}$	16.41 ± 1.84 a	18.50 ± 1.35 a
Procyanidin A2	$1.38 \pm 0.03 \mathrm{b}$	$1.34 \pm 0.13 \mathrm{b}$	$1.42 \pm 0.04 \mathrm{b}$	1.54 ± 0.05 a
Procyanidin B1	$9.56 \pm 0.34 \mathrm{b}$	$11.60\pm2.74~\mathrm{ab}$	$11.62 \pm 0.43 \text{ ab}$	12.57 ± 1.04 a
Procyanidin B2	6.56 ± 0.14 a	6.27 ± 0.54 a	$5.43 \pm 0.18 \mathrm{b}$	$5.31 \pm 0.29 \mathrm{b}$
∑ Flavan-3-ols	64.26 mg/L	64.29 mg/L	74.70 mg/L	80.59 mg/L
Flavonols				
Kaempferol-3-O-glucoside	$2.81 \pm 0.04 \mathrm{d}$	5.89± 0.53 c	$7.08 \pm 0.14 \mathrm{b}$	7.56 ± 0.22 a
Quercetin-3-O-β-D-glucoside	$46.13 \pm 0.10 \mathrm{c}$	$95.16 \pm 5.87 \mathrm{b}$	106.59 ± 0.87 a	$107.87 \pm 3.41~a$
Isorhamnetin-3-O-glucoside	$25.62 \pm 0.54 \mathrm{c}$	$39.60 \pm 2.16 \mathrm{b}$	43.15 ± 0.48 a	43.08 ± 1.34 a
Myricetin	0.98 ± 0.05 a	1.00 ± 0.12 a	0.93 ± 0.05 a	$0.97 \pm 0.06 a$
Rutin	0.65 ± 0.10 a	0.72 ± 0.10 a	0.69 ± 0.03 a	0.71 ± 0.08 a
∑ Flavonols	76.19 mg/L	142.37 mg/L	158.44 mg/L	160.19 mg/L
∑ Phenolic compounds by HPLC-DAD-FD	233.39 mg/L	412.04 mg/L	419.08 mg/L	414.68 mg/L

 $^{^{\}rm a}$ Results are expressed as mean values \pm standard deviation. Values followed by different letters indicate a significant difference between the samples according to Tukey's test (p \leq 0.05). $^{\rm b}$ TW: Traditional winemaking. TV55: Thermovinification at 55 °C. TV65: Thermovinification at 65 °C. TV75: Thermovinification at 75 °C.

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Five anthocyanins were quantified in the red wines. The TV75 wine exhibited the lowest concentrations for this class of compounds, suggesting that the higher temperature (\geq 75 °C) led to a greater degradation of anthocyanins. Compared to the control treatment (TW), TV65 wines lost approximately 28.72% of their total anthocyanin content, whereas in TV75, the decrease in these compounds was approximately 58.78%. Geffroy et al. [20] evaluated the use of thermovinification in Carignan grape musts (temperatures of 50 °C and 75 °C for 30 min and 3 h) and observed an increase in anthocyanins when the must was heated at 50 °C for 3 h and a decrease in anthocyanins through thermovinification at 75 °C for 3 h. This study showed that the use of thermovinification at 55 °C for 2 h significantly favoured the extraction of pelargonidin-3-O-glucoside and delphinidin-3-O-glucoside but not of malvidin-3-O-glucoside, petunidin-3-O-glucoside, or peonidin-3-O-glucoside.

Malvidin-3-O-glucoside was the most abundant anthocyanin, as previously reported for red wines [36], and was present at higher concentrations in TW wine (30.61 mg/L). However, this wine did not differ significantly (p < 0.05) in the malvidin-3-O-glucoside content from the wine elaborated by thermovinification at 55 °C (TV55). Orbanic et al. [24], analysing wines from Teran whose grapes underwent pre-fermentation heating (50 °C for 48 h), observed that malvidin-3-O-glucoside represented 70% of the total anthocyanin concentration. They reported values similar to those found in this study for the TW and TV55 samples (27.77 mg/L).

Regardless of the temperature, the content of phenolic acids decreased due to the influence of thermovinification, except *trans*-caftaric acid, which was approximately 50 times higher in the treatments subjected to thermovinification (ranging from 119.07 mg/L in TV75 to 132.17 mg/L in TV55) compared to traditional winemaking (2.64 mg/L). Transcaftaric acid is a hydroxycinnamic acid found in high quantities in grapes and wines [37], and was the most abundant phenolic compound found in this study. Rossi et al. [35] and Lukić et al. [38] also observed a significant increase in some hydroxycinnamic acids (*trans*-caftaric and ferulic acids) after 48 h of pre-fermentative maceration heating at 45 °C (plus eight days of traditional maceration) and 50 °C for 6 h, respectively. Rossi et al. [35] reported a trans-caftaric acid level of 79.65 mg/L, whereas Lukic et al. [38] found a level of 137.35 mg/L. Although hydroxycinnamic acids do not directly influence the flavour of wines, they are precursors of volatile phenols and exhibit antimicrobial and antioxidant activities [39].

Concerning the stilbenes identified in Syrah wines, thermovinification had a remarkable impact on the extraction of many individual stilbenes, and on the total stilbene content. Three stilbenes were identified, *cis*-resveratrol, piceatannol, and trans-resveratrol, and they were found at higher levels in the treatment subjected to 65 °C (TV65), with a total concentration of 0.18, 2.28, and 0.62 mg/L, respectively. These results are in line with those found by Orbanic et al. [24], where the pre-fermentative heating maceration treatment (50 °C for 48 h) led to a significant increase in the concentrations of total and individual stilbenes (piceatannol, trans-resveratrol, cis-piceid, and trans-piceid). The major dietary sources of stilbenes in humans are grapes and wine [40].

The non-flavonoid groups of phenolic compounds include hydroxybenzoic acids, hydroxycinnamic acids, volatile phenols, and stilbenes, which are recognised for their ability to enhance and stabilise the colour of red wines through intra- and inter-molecular reactions [41]. Furthermore, they are recommended for the sensory profile of wine, especially the volatile compounds generated by phenolic acids and stilbenes, such as resveratrol, which can carry out potent biological activities that have beneficial effects on human health [24,41]. This highlights the importance of using thermovinification to obtain wine rich in these compounds. In addition to promoting greater beverage stability, this technique can enhance nutraceutical quality.

For the class of flavan-3-ols, seven compounds were quantified, and the TV75 treatment stood out, particularly in (+)-catechin (35.01 mg/L), (-)-epicatechin (18.50 mg/L), procyanidin A2 (1.54 mg/L), and procyanidin B1 (12.57 mg/L). Compared to another emerging maceration technique, such as carbonic maceration, the results in this study

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were higher than those reported by Tong et al. [42] with Cabernet Sauvignon wines. They found concentrations of 12.21, 4.33, and 5.34 mg/L for (+)-catechin, (-)-epicatechin, and procyanidin B1, respectively. Orbanić et al. [24], when subjecting Teran grapes to heating at 50 °C for 48 h, also found in the wine minor values for (+)-catechin (33.56 mg/L) and (-)-epicatechin (14.33 mg/L) compared to TV75.

Owing to their antioxidant properties, flavan-3-ols play a key role in the beneficial health properties related to moderate wine consumption. Therefore, a considerable amount of literature has been published on their biological activities, including their anticarcinogenic, antidiabetic, cardioprotective, or neuroprotective effects [43]. With the use of thermovinification at 65 °C and 75 °C, an increase in this class of compounds by 16.26% and 25.42%, respectively, was observed compared to the control treatment. Wine phenolic composition is greatly influenced by many factors, including vineyard environmental conditions, winemaking practises, and wine storage [44]. Thus, thermovinification as a maceration technique at the temperatures and times used in the present study could contribute to the production of Syrah wine with greater antioxidant potential.

Among the flavonols, kaempferol-3-O-glucoside, quercetin-3- β -D-glucoside, and isorhamnetin-3-O-glucoside were higher in the TV65 and TV75 wines, while myricetin and rutin showed no significant differences (p < 0.05) between all the treatments. Flavonols can stabilise colours by participating in copigmentation reactions with anthocyanins [37] and are part of the phenolic compound group recognised for their antioxidant, anti-carcinogenic, anti-inflammatory, and immune functions in humans against coronavirus infection (SARS-CoV-2) and other diseases [45]. When compared to the control treatment, thermovinification at 65 °C and 75 °C over a period of 2 h allowed for an increase of 107.92% and 110.23%, respectively, in the total class of flavonols in the Syrah wine.

3.2. Colorimetry

The results of the colorimetric analyses (CIELab and CIEL*C*h systems) of the wines are shown in Figure 3. The use of thermovinification reduced Syrah wine luminosity (L*), making it darker, with the reduction in luminosity being proportional to the increase in temperature, ranging from 13.02 in TV75 to 14.83 in TV55. When analysing the luminosity of Cabernet Sauvignon wines subjected to thermovinification ($70 \, ^{\circ}\text{C}/30 \, \text{min}$), El Darra et al. [46] found a value of 19.70; however, it did not differ significantly in relation to the control (23.35).

The red colour coordinate (a*) was also affected by the thermovinification practice, decreasing as the temperature increased to more than 65 $^{\circ}$ C. This reduction in the red colour may be associated with the degradation of anthocyanins, particularly in the treatment using 75 $^{\circ}$ C (TV75).

For the yellow colour coordinates (b*), wines from all treatments showed significant differences (p < 0.05). The control treatment (TW) had the lowest yellow colour intensity (4.48). This may be linked to the lower concentration of total flavonols found in wines from this treatment (76.20 mg/L) (Table 1). Flavonols are yellow pigments typically found in their glycosidic form. The most reported flavonols in wines are myricetin, kaempferol, isorhamnetin, and quercetin [47]. Therefore, it can be inferred that thermovinification can improve the flavonol content of Syrah wine and consequently increase the intensity of the yellow colour. Wines subjected to thermovinification had total flavonol contents of 142.37, 158.43, and 160.18 mg/L in TV55, TV65, and TV75, respectively. Additionally, considering the hue angle (h°), the wines appeared at intermediate points (48.06°, 49.46°, 51.41°, and 67.19° for TW, TV55, TV65, and TV75, respectively) between the red (0°) and yellow (90°) colours. TV75 wine showed the angle closest to the b* coordinate (yellow), differing significantly (p < 0.05) from the other treatments.

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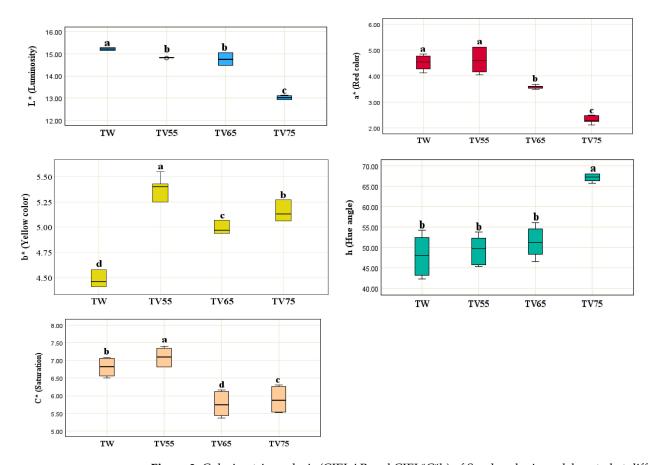


Figure 3. Colorimetric analysis (CIELAB and CIEL*C*h) of Syrah red wines elaborated at different thermovinification temperatures. Samples: TW, Traditional winemaking; TV55, Thermovinification at 55 °C; TV65, Thermovinification at 65 °C; and TV75, Thermovinification at 75 °C. Colour coordinates: L*, luminosity; a*, red/green component; b*, blue/yellow component; C*, saturation; and h, hue angle (h) Different letters indicate a significant difference between samples according to Tukey's test ($p \le 0.05$).

3.3. Principal Component Analysis (PCA)

PCA was performed to explain the influence of the thermovinification temperature on the phenolic composition and colour of Syrah wines (Figure 4). The first principal component (PC1), which explained 69.39% of the sample variability, was responsible for separating the wines from the control treatment (TW) and TV55 from the wines from TV65 and TV75. This indicates that wines from TW and TV55 were more similar to each other, just as wines from TV65 and TV75 were more similar to each other.

The wines TW and TV55 were found in the negative part of PC1 and may be associated with higher concentrations of ρ -coumaric, ferulic, chlorogenic, gallic, and caffeic acids; peonidin-3-O-glucoside; delphinidin-3-O-glucoside; pelargonidin-3-O-glucoside; malvidin-3-O-glucoside; petunidin-3-O-glucoside; (—)—epicatechin gallate; (—)—epigallocatechin gallate; procyanidin B2; and the colour components L*, a*, and C*, converging with the results found in Table 1 and Figure 3. Traditional vinification and thermovinification at a lower temperature (55 $^{\circ}$ C) can result in wines with similar concentrations of the compounds mentioned above, as well as similarities in L*, a*, and C*.

In the opposite position, located in the positive part of CP1, are the wines TV65 and TV75. The vectors of trans-resveratrol, piceatannol, *cis*-resveratrol, *trans*-caftaric acid, rutin, isorhamnetin-3-O-glucoside, kaempferol-3-O-glucoside, quercetin-3- β -D-glucoside, (–)-epicatechin, (+)-catechin, procyanidins A2 and B1, and the h were closer to these wines. These compounds and h were responsible for the similarity between the samples TV65 and

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TV75, suggesting that the thermovinification at temperatures above 65 $^{\circ}$ C favoured the greater extraction of stilbenes, flavonols, and flavan-3-ols.

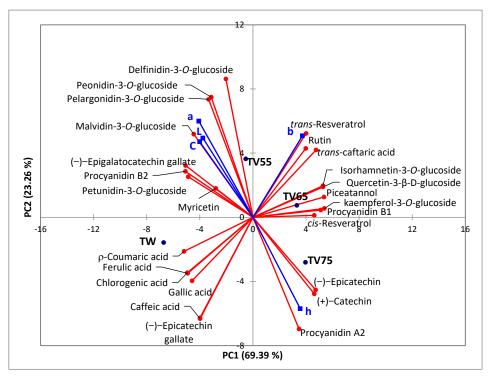


Figure 4. The principal component analysis (PCA) showing the configuration of Syrah wines vinified using different thermovinification temperatures, phenolic compounds (n = 26 compounds), and colour coordinates (L*, a*, b*, C*, and h). Samples: TW, Traditional winemaking; TV55, Thermovinification at 55 °C; TV65, Thermovinification at 65 °C; and TV75, Thermovinification at 75 °C.

3.4. Sensory Analysis—Consumer Test

The results of the sensory analysis are shown in Figure 5. The consumer test was conducted solely with the wines thermovinified at 65 °C (TV65). The exclusive sensory analysis of the 65 °C thermovinification wine is justified by its superior quality characteristics. This thermovinification temperature at 65 °C during 2 h promoted the preservation of important bioactive compounds and the lower degradation of anthocyanins, which are very important to young red wine's sensory characteristic.

Figure 5A shows the data from the acceptance means using a nine-point hybrid hedonic scale. Wine appearance had the highest score (mean score: 7.5), followed by aroma, overall impression, and flavour (mean scores: 6.8, 6.7, and 6.3, respectively). These data indicated a good prospect for thermovinification Syrah wines. Additionally, the largest percentage of consumers (53.4%) expressed a positive intention to buy the 65 °C-thermovinified wine (21.7% would definitely buy, and 31.7% would possibly buy), while 26.7% of respondents indicated no interest in purchasing (Figure 5B).

The practice of thermovinification at $65\,^{\circ}$ C for 2 h presents as a good alternative for producing young red wines with high market potential, aligning with current trends for beverages that offer more functional benefits. Thus, the consumer test result contributed to reinforce the relevance of thermovinification practice, showing its importance to the wine industry.

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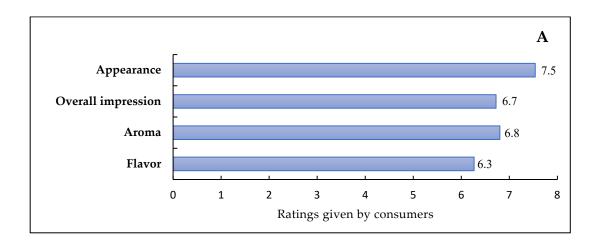




Figure 5. Data obtained from the sensory analysis of the Syrah thermovinified red wine at 65 °C. **(A)** The acceptance test conducted using a 9-point hybrid hedonic scale. **(B)** Results of the purchase intention test.

4. Conclusions

Significant changes in the profile of phenolic compounds and the colouration of Syrah wines were observed with the application of thermovinification for 2 h, notably using temperatures above $65\,^{\circ}$ C. The 2 h period is recommended in the literature for thermovinification.

Bearing in mind that the profile of phenolic compounds in wines subjected to thermovinification at a temperature of 55 $^{\circ}$ C resembled that of wines produced by traditional vinification (control) and that when subjected to thermovinification at 75 $^{\circ}$ C, there was a degradation of anthocyanins, which is an important class of phenolic compounds for red wines, it can be stated that the temperature of 65 $^{\circ}$ C would be the most appropriate owing to the greater extraction of some phenolics and the less degradation of anthocyanins.

The results obtained are related to microvinifications (10-litre glass) and demonstrate that thermovinification is an excellent alternative for the production of Syrah wines as a substitute for conventional maceration. Thermovinification is an alternative technique that enhances the stability, sensory acceptability, and nutraceutical potential of young red wines. These characteristics align with the growing consumer interest in high-quality red wine, which has potential health benefits. Further studies are needed to better understand the impact of thermovinification on the volatile compound profile and descriptive sensory characteristics of red wine.

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