

INFLUENCE OF IRRIGATION STRATEGIES ON THE PHYSICOCHEMICAL PROPERTIES OF 'SYRAH' WINE PRODUCED IN SÃO FRANCISCO VALLEY

VANESSA DE SOUZA OLIVEIRA¹; GIULIANO ELIAS PEREIRA²; AUGUSTO MIGUEL NASCIMENTO LIMA¹; ALESSANDRA MONTEIRO SALVIANO³; RUSSAIKA LÍRIO NASCIMENTO¹ E LUÍS HENRIQUE BASSOI⁴

¹Univasf, Colegiado de Engenharia e Ambiental, Avenida Antonio Carlos Magalhães, 510, 48902-300, Juazeiro - BA, Brasil. vanessa.soliveira@gmail.com, augusto.lima@univasf.edu.br, nrussaika@gmail.com

²Embrapa Uva e Vinho, Rua Livramento, 515, Caixa Postal 130, 95701-008, Bento Gonçalves - RS, Brasil. giuliano.pereira@embrapa.br

³Embrapa Semiárido, BR 428 km 152, Caixa Postal 23, 56302-970, Petrolina - PE, Brasil. alessandra.salviano@embrapa.br

⁴Embrapa Instrumentação, Rua XV de Novembro, 1452, Caixa Postal 741, 13560-970, São Carlos - SP, Brasil. luis.bassoi@embrapa.br

1 ABSTRACT

Deficit irrigation is used to control grapevine vegetative vigor and the grape composition can be influenced by soil water content. The minerals contained in the grapes are responsible for the physicochemical properties and stability of the wine. Therefore, the aim of this work was to evaluate the effects of irrigation strategies on the properties of 'Syrah' wines produced in São Francisco Valley, Brazil. The irrigation treatments used were FI (full irrigation), RDI (regulated deficit irrigation), and DI (deficit irrigation). Winemaking was performed by traditional methods, followed by stabilization and bottling. Density, alcohol content, pH, total and volatile acidity, total and free sulfur dioxide, total polyphenols index, color intensity, and total anthocyanin content were determined, as well as the contents of Ca, Mg, P, K, Na, Mn, Fe, Cu, Zn, Cd, Cr, Ni, and Pb. The properties of wines were influenced by irrigation strategies, except for the content of total anthocyanin, Mg, P, Na, Cu, and Mn. The contents of Cd, Cr, Fe, and Zn were similar in the vines. The wines demonstrated varied properties with different typicalities. Irrigation strategies can be recommended for different products, FI for young and cheap, and DI and RDI for aged and more expensive wines.

Keywords: *Vitis vinifera* L., grape, water availability, tropical wine.

OLIVEIRA, V. DE S.; PEREIRA, G. E.; LIMA, A. M. N.; SALVIANO, A. M.;
NASCIMENTO, R. L. E BASSOI, L. H.
INFLUÊNCIA DE ESTRATÉGIAS DE IRRIGAÇÃO NAS PROPRIEDADES FÍSICO-
QUÍMICAS DO VINHO 'SYRAH' PRODUZIDO NO VALE DO SÃO FRANCISCO

2 RESUMO

A irrigação com déficit é utilizada para controlar o vigor vegetativo da videira, e a composição da uva pode ser influenciada pela umidade do solo. Os minerais presentes nas uvas são responsáveis pelas propriedades físico-químicas e pela estabilidade do vinho. Portanto, o objetivo deste trabalho foi avaliar os efeitos de estratégias de irrigação sobre as propriedades do vinho 'Syrah' produzido no Vale do São Francisco, Brasil. As estratégias de irrigação utilizadas foram IP (irrigação plena), IDC (irrigação com déficit controlado) e ID (irrigação com déficit). A elaboração de vinhos foi realizada por métodos tradicionais, seguido de estabilização e engarrafamento. A densidade, teor de álcool, pH, acidez total e volátil, dióxido de enxofre total e livre, o índice total de polifenóis, a intensidade da cor, conteúdo total de antocianina e concentrações de Ca, Mg, P, K, Na, Mn, Fe, Cu e Zn, Cd, Cr, Ni e Pb foram determinadas. As propriedades dos vinhos foram influenciadas pelas estratégias de irrigação, exceto os teores totais de antocianinas, e de Mg, P, Na, Cu e Mn. Os níveis de Cd, Cr, Fe e Zn foram semelhantes nos vinhos. Os vinhos demonstraram propriedades variadas com diferentes tipicidades. As estratégias de irrigação podem ser recomendadas para a obtenção de diferentes produtos, sendo a IP para vinhos jovens e baratos, e o ID e IDC para vinhos envelhecidos e mais caros.

Palavras chave: *Vitis vinifera* L., uva, disponibilidade de água, vinho tropical.

3 INTRODUCTION

The use of irrigation in viticulture is a common practice in warm and dry regions, such as the Northeast region of Brazil, and it is an efficient way to regulate the availability of water and nutrient to the grapevines. This enables the growth of grapes with distinguished qualities that can be used for winemaking (ETCHEBARNE; OJEDA; DELOIRE, 2009). Wine quality is directly associated with the grape composition and the winemaking process (GARCÍA-ESTÉVEZ et al., 2015).

Grapes used for winemaking are cultivated under conditions of stress in order to obtain better quality wines which affect the nutrient availability to the vines (OLLÉ et al., 2011; PEYNAUD, 1997). Monitoring the water deficit during vine cultivation, especially during the maturation of the grape, is important for the synthesis and the increase in the concentration of beneficial compounds, the reduction of acidity, and the accumulation of aromatic and phenolic compounds. The

latter are responsible for the sensory characteristics of the wine (color, aroma, and structure), color stability, and wine-aging ability (SANTESTEBAN; MIRANDA; ROYO, 2011). The amount of water that is available to the vine depends on the purpose of grapes cultivation (table grapes or winemaking) as well as on the type of wines to be elaborated and market niches, young wines with a light structure or aging wines with an intense tannin structure (REYNIER, 2007; PEYNAUD, 1997).

The mineral composition of the wine contributes to its stability and sensory characteristics, influencing wine color and clarity. Cations and anions are naturally present in the grape juices and grape wines at non-toxic levels. Heavy metals are mineral elements that are also present in the wine, and even in small concentrations, they can negatively affect wine quality. The practice of assessing the heavy metals present in wines is related to the quality of the product, considering that such ions may cause instability and alterations in the

sensory characteristics due to chemical interactions. These heavy metals are also known to be associated with toxic functions in humans (RIBÉREAU-GAYON et al., 2003).

Despite the importance of the Lower Middle region of the São Francisco Valley in the production of excellent quality wines, which supply the internal and external markets, studies that aim to evaluate the effects of water availability on mineral content of the grapes, as well as its relation to the physicochemical properties of wines, are limited. In this context, the present work aims to evaluate these properties of the wines obtained from grapevine 'Syrah' subjected to different irrigation strategies.

4 MATERIAL AND METHODS

The experiment was carried out in a vineyard installed at the Experimental Field of Bebedouro, Embrapa Tropical

Semi-arid, located in Petrolina, State of Pernambuco, Brazil, latitude 9° 08'08,9''S, longitude 40°18'33,6''O and altitude of 373 m. According to the Geoviticulture Multicriteria Climatic Classification System, this region shows type DII HI6 CII climate, with moderate dryness, high temperatures, and warm nights. Annually, the average climate variables were as follows: air temperature of 26.5°C, precipitation of 541.1 mm, relative air humidity of 65.9%, class A pan evaporation of 2,500 mm year⁻¹, and wind velocity of 2.3 m s⁻¹. The rainfall is irregularly distributed, concentrated between December and April, and the annual insolation is higher than 3,000 h (VELEDA et al., 2015). The soil of the area was classified as Eutrophic Red-Yellow Argisol (SANTOS et al., 2018) or Ultisol (SOIL SURVEY STAFF, 2014), presenting the physicochemical properties shown in Table 1. Soil analyses were performed following the procedures described by Donagema et al. (2011).

Table 1. Physicochemical properties of the Eutrophic Red-Yellow Argisol (Ultisol) cultivated with grapevines.

Depth	d _b	d _p	Sand	Silt	Clay	pH H ₂ O	ECe	H+Al
M	--kg dm ⁻³ --		-----g kg ⁻¹ -----			1:2,5	dS m ⁻¹	cmol _c dm ⁻³
0-0.10	1.32	2.53	765.38	123.94	106.73	6.79	0.38	1.29
0.10-0.20	1.37	2.53	756.70	112.40	130.89	6.57	0.32	1.62
0.20-0.40	1.35	2.51	716.93	113.66	169.40	6.41	0.21	1.59
0.40-0.60	1.32	2.52	746.86	140.78	112.35	6.27	0.28	1.74
	BS	CEC	OM	P	K	Ca	Mg	Na
	cmol _c dm ⁻³		g kg ⁻¹	mg dm ⁻³		-----cmol _c dm ⁻³ -----		
0-0.10	7.80	9.03	27.41	113.25	0.52	4.80	2.65	0.09
0.10-0.20	6.50	8.04	12.90	96.64	0.53	3.74	2.47	0.08
0.20-0.40	5.70	7.50	7.32	91.85	0.34	3.32	2.22	0.06
0.40-0.60	5.30	6.99	5.69	54.90	0.29	3.03	1.98	0.06

d_b = bulk density; d_p = particle density; ECe = electrical conductivity of the saturation past; BS = base sum; CEC = cation exchange capacity; OM = organic matter.

'Syrah' was the cultivar studied, grafted to the rootstock 1103 Paulsen. The grapevines were planted on the 30th of April 2009, with 3 m spacing between rows, and 1 m between plants. During

planting, the fertilizing was performed based on soil analyses (Table 1) with lime (200 g per linear meter), goat manure (40 m³ ha⁻¹), and granular simple super phosphate (18% P₂O₅) (50 g plant⁻¹). The

fertilizing used during productive cycle was only goat manure at a rate of $0.02 \text{ m}^3 \text{ plant}^{-1}$ (application into the soil in the beginning of the growing season). The plant trellis system used was espaliers with double spur cordon with upward-slanting branches. The period of vineyard formation (vegetative growth) occurred until April 13rd 2010, when pruning was performed for the first productive cycle. This study was performed in the third one, from May 10th to September 8th, 2011. The drip irrigation system was used with emitters spaced at 0.5 m intervals in the plant rows, and with a flow of 2.5 L h^{-1} , with a pressure of 100 kPa, measured by a field flow test. Irrigation management was conducted according to the crop evapotranspiration (ET_c, mm), determined by multiplying the reference evapotranspiration (ET_o, mm), and the crop coefficient (kc) for the phenological stages of wine grapevines as obtained by da Basso et al. (2007). The ET_o was predicted by using the Penman-Monteith FAO method and the measured parameters registered by the automatic agrometeorological station installed 60 m away from the experiment site. During the experiment, the chemicals used in pest and disease controls in the area were as follows: Equation® and Forum® for mildew control, at a dose of $100 \text{ g } 100 \text{ L}^{-1}$ (two applications); Rumo® for borer control at a dose of $8 \text{ g } 100 \text{ L}^{-1}$ (one application); Rubigam® for rust control at a dose of $35 \text{ g } 100 \text{ L}^{-1}$ (one application); and Amistar® and Cabrio top® for powdery mildew control at a dose of $60 \text{ g } 100 \text{ L}^{-1}$ (one application).

Each experimental plot contained 48 plants, set out in two rows of 24. The experimental design used was randomized blocks with three treatments and four replicates. The treatments comprised three irrigation strategies: FI (full irrigation, where irrigation was performed to replace the amount of water lost by ET_c, during

the productive cycle); RDI (regulated deficit irrigation), where the water was supplied intermittently from the phenological phase of bunch closure (48 days after pruning - DAP), but conducted at 70, 71, 87, 90, 111 DAP, to increase soil moisture (θ , $\text{m}^3 \text{ m}^{-3}$) in the effective depth of the root system (0.6 m). This was performed according to the soil moisture conditions monitored weekly by the neutron moderation technique, at depths of 0.15, 0.30, 0.45, 0.60, 0.75, 0.90, 1.05, and 1.20 m; and DI (deficit irrigation), where the water application was interrupted from the 48 DAP until the grape harvesting, at 121 DAP.

During harvest, three grape bunches from all three irrigation treatments were randomly sampled and weighted. Bunches were sent to the Enology Laboratory at Embrapa Tropical Semi-Arid and maintained in a cold chamber at 10°C for 24 h, avoiding premature oxidation and spoilage due to the high temperature. Then, grapes were processed.

Winemaking was performed in triplicates by the traditional method (PEYNAUD, 1997), and 27 kg of grapes were processed for each irrigation treatment (FI, RDI, and DI). Initially, the grapes were de-stemmed and slightly crushed, with addition of potassium metabisulphite ($50 \text{ mg } \text{L}^{-1}$) to avoid premature oxidation. The must was then transferred into glass tanks, and $20 \text{ g } 100 \text{ L}^{-1}$ of *Saccharomyces cerevisiae* were added in a controlled temperature room at 25°C for the alcoholic fermentation. The density and temperature of the must during winemaking were daily monitored, as well as two pumping over per day to aerate and promote yeast proliferation. Once a density below 0.993 was reached, which coincided to the end of alcoholic fermentation at fifth day, wines were pressed to remove skins and seeds, and then transferred to another tank with controlled temperature room at 18°C for the malolactic fermentation. After

30 days, the wines were stabilized in a cold chamber at 0°C for 30 days. After this time, sulphur dioxide (SO₂) was added for wine corrections to 40 mg L⁻¹ (free SO₂), and then wines were bottled and stored horizontally in a controlled temperature room at 18°C. Wines were analyzed 30 days after bottling. To characterize the physicochemical properties and mineral and heavy metal contents of the wines, three bottles from each treatment were used and the analyses were carried out also in triplicates.

The physicochemical analyses consisted of determining the pH, total titratable acidity, alcoholic content, density, dry extract, volatile acidity, total and free SO₂, total polyphenols index (TPI), color intensity, and total anthocyanins of the wines, according to the procedures proposed currently by Office International de la Vigne et du Vin (2016). Wines were directly analyzed without any dilution or filtration, by the routine analyses listed before and normally used.

The K and Na contents were determined by flame emission photometry. The Ca, Mg, Mn, Fe, Cu, and Zn contents were determined by atomic absorption spectrophotometry (AAS). Contents of the heavy metals Cd, Cr, Ni, and Pb were determined by inductively coupled plasma-optical emission spectrometry ICP-OES (DUTRA et al., 2011).

The analysis of variance (ANOVA) was performed and the average of the

physicochemical properties were compared by Tukey test at 5%. To complement the evaluation of the results, the principal component analysis (PCA) was performed (ZIÓŁKOWSKA; WĄSOWICZ; JELEŃ, 2016).

5 RESULTS AND DISCUSSION

5.1 Weight of bunch

The full irrigation (FI) treatment resulted in a higher individual mass of bunches per plant (150 g), which differed from the RDI (120 g) and DI (110 g) treatments. There were no differences between the last two treatments. Under water stress, the grapes were normally smaller due to lower availability of water for cellular elongation (ROMERO et al., 2016), which reflected in the lower weight of bunches. Bassoi et al. (2011) showed that the water deficit generally results in smaller grapes and modifies the mineral composition of the fruit, then corroborating the results from this study.

5.2 Wine physicochemical properties

The results from the wines are showed in Table 2. The values obtained for total anthocyanins, total SO₂ and free SO₂ content were not significantly different (PEYNAUD, 1997).

Table 2. Physicochemical properties of wines obtained from grapevines 'Syrah' subjected to full irrigation (FI), regulated deficit irrigation (RDI) and deficit irrigation (DI) treatments.

Parameters	Treatments		
	FI	RDI	DI
Density	0.99 ± 0.00a	0.99 ± 0.00ab	0.99 ± 0.00b
Alcohol (%v/v)	13.90 ± 0.40b	15.39 ± 0.34a	15.76 ± 0.22a
pH	3.90 ± 0.00a	3.83 ± 0.06ab	3.80 ± 0.00b
Total acidity (g L ⁻¹)	4.80 ± 0.52b	5.25 ± 0.26ab	5.85 ± 0.00a
Volatile acidity (g L ⁻¹)	0.71 ± 0.07ab	0.66 ± 0.00b	0.80 ± 0.03a
Total SO ₂ (mg L ⁻¹)	36.69 ± 5.91a	45.22 ± 5.91a	44.37 ± 7.39a
Free SO ₂ (mg L ⁻¹)	25.60 ± 1.28a	23.89 ± 2.96a	23.89 ± 1.48a
Total polyphenols index (I 280)	49.03 ± 2.36b	55.56 ± 2.80a	60.00 ± 0.72a
Color intensity	4.64 ± 0.65b	6.13 ± 0.20a	7.18 ± 0.42a
Total anthocyanins (mg L ⁻¹)	513.58 ± 30.66a	560.53 ± 53.07a	532.98 ± 13.30a

Rows followed by the same letter are not different at 5% probability (Tukey).

Conversely, significant differences ($p < 0.05$) were found among the treatment in the following parameters: density, alcohol, pH, TPI, color intensity, total and volatile acidity. For the density and pH, the higher values occurred with the FI treatment in comparison to the DI treatment, whereas a greater amount of alcohol, TPI and color intensity were observed with the RDI and DI treatments (Table 2).

The pH of the wines from the FI treatment was higher than that from the DI, presenting means of pH 3.9 and 3.8, respectively. These values are considered high since a pH between 3.3 and 3.6 is desirable for red wines, according to the studies performed by Peynaud (1997), in temperate climates. In tropical regions, the pH of wines is higher, as shown by Padilha et al. (2017), who suggested that this is due to the higher uptake of minerals by the increased supply of water. In general, the wines with pH values equal to or higher than 3.9 are more susceptible to oxidation, presenting loss of fresh aromas, change in color and least shelf life (RIBÉREAU-GAYON et al., 2003).

The alcohol contents were 15.76

°GL, obtained from the DI treatment, and 15.39 °GL, from RDI (Table 2). These values are above those established by the Brazilian and OIV legislations (OFFICE INTERNATIONAL DE LA VIGNE ET DU VIN, 2016; BRASIL, 2014). The allowed interval varies from 8.6 to 14 °GL for table wines (BRASIL, 2004). The climatic conditions of this region shorten the maturation period due to high solar radiation indexes, insolation, and air temperature, and reduce the accumulation of sugar in the grapes. On the other hand, the increased sugar concentration was due to the dehydration in some of the grapes in the bunch, and the reduced bunch weight was a result of the lower water supply by the treatments DI and RDI. This is a consequence of the increased skin/pulp ratio, which is favored by a low availability of water for cellular elongation (ROMERO et al., 2016; PEYNAUD, 1997). The treatments that led to lower weight in grape bunches (DI and RDI) were those that had higher alcohol values in the wines (Table 2), whereas the FI treatment provided higher weight values and lower alcohol content.

The total acidity values found in the

present work (Table 2) are within the values established by legislation, which allows variation between 4.12 and 9.75 g L⁻¹ of tartaric acid (BRASIL, 2004). The highest total acidity value was found in the DI treatment (5.85 g L⁻¹), whereas the lowest value was found in the FI treatment (4.80 g L⁻¹). Volatile acidity corresponds to fatty acids of the acetic series that are found in wines. This value indicates the sanitary state and severity of some microbiological alterations that occur in wines. From a qualitative point of view, lower values are ideal (RIBÉREAU-GAYON et al., 2003; PEYNAUD, 1997). The volatile acidity values for all the treatments evaluated were below those established by the Brazilian legislation, i.e., a maximum 20 mEq L⁻¹ or 1.2 g L⁻¹ (BRASIL, 2004).

The mean contents of total and free SO₂ varied from 23.89 mg L⁻¹ to 25.60 mg L⁻¹, and 36.69 mg L⁻¹ to 45.22 mg L⁻¹, respectively. These values are considered ideal to ensure protection against oxidation during the winemaking, as well as stability after bottling. Values depend mainly on the health status of grapes at harvest.

The total polyphenols index (TPI) is an analytical and enological parameter that represents the group of compounds present in wines, particularly the visual and structural characteristics, and the sensory and antioxidant properties, then highlighting the role of anthocyanins and tannins (GARCÍA-ESTÉVEZ et al., 2015). The higher TPI values were obtained with the DI (60.00) and RDI (55.56) treatments, indicating that the irrigation strategies with deficit promoted a higher synthesis of polyphenols, which may have influenced the tannins (flavanols), phenolic acids, or even some of the anthocyanins present in the wine (RIBÉREAU-GAYON et al., 2003; USSEGLIO-TOMASSET, 1995). A similar trend was observed for the color intensity values (Table 2), obtained by the sum of absorbance at 420, 520, and 620

nm, which represent the colors yellow, red, and blue, respectively.

The irrigation strategies produced grapes and wines with distinct properties among the treatments evaluated. The treatments comprising hydric stress resulted in a higher specific superficial area in the grape skin (lower weight), depending on the higher number of smaller grapes. Specific superficial area is important in the extraction of polyphenols retained in the skin during the maceration time at alcoholic fermentation (GARCÍA-ESTÉVEZ et al., 2015). This can explain the higher TPI content found for RDI and DI treatments in this study. Similar results were found by Ollé et al. (2011) from wines elaborated from grapevines under DI treatment, followed by those under RDI and FI treatments. The last one presented lower values of TPI.

In the present study, no statistical differences were found for total anthocyanins, but it was observed that the DI and RDI treatments provided values of higher magnitude (Table 2). On the other hand, Reynier (2007) and Niculcea et al. (2015) stated that the presence of anthocyanins in the must is influenced by irrigation. Furthermore, Peynaud (1997) and Ribéreau-Gayon et al. (2003) also reported an increase in the total anthocyanin and phenol content, due to the reduction level of irrigation for different cultivars evaluated, including ‘Cabernet Franc’ and ‘Syrah’.

5.3 Mineral Composition

The Ca content in wines is usually between 60 and 110 mg L⁻¹ (DUTRA et al., 2011). The Ca values found in this study was below this range for all the evaluated treatments, showing similar values among treatments, with a minimum of 48.46 mg L⁻¹ and a maximum of 52.74 mg L⁻¹ (Table 3). The knowledge of the Ca content in wines is important, since it is

associated with the precipitation of calcium tartrate, which occurs slowly and, generally, after bottling, and this may

influence the increase of pH in wines, decreasing the stability and affecting product preservation.

Table 3. Mineral composition of wine from 'Syrah' grapevines subjected to full irrigation (FI), regulated deficit irrigation (RDI) and deficit irrigation (DI).

Elements (mg L ⁻¹)	Treatments		
	FI	RDI	DI
Ca	48.46a	52.74a	48.46a
Mg	95.18b	100.47a	95.14b
P	97.67a	85.67b	70.68c
K	1796.75a	1789.43a	1804.07a
Na	8.23c	26.06a	15.64b
Cu	0.32a	0.13b	0.26ab
Fe	0.88a	0.85a	0.72a
Mn	1.79b	2.12a	1.84b
Zn	0.61a	0.60a	0.59a
Cd	0.04a	0.05a	0.05a
Cr	0.02a	0.01a	0.01a

Rows followed by the same letter are not different at 5% probability (Tukey).

The Mg content in wines from the RDI treatment was higher when compared to the FI and DI treatments, with 100.47 mg L⁻¹, 95.18 mg L⁻¹, and 95.14 mg L⁻¹, respectively, which are in agreement with the values described in the literature varying from 80 mg L⁻¹ to 120 mg L⁻¹ (DUTRA et al., 2011). Pereira et al. (2007), when evaluating the wines from the Northeast of Brazil, found Mg contents within the range 56.5 mg L⁻¹ to 130.9 mg L⁻¹. On the other hand, Rizzon, Salvador and Miele (2008) found values between 50 mg L⁻¹ and 90 mg L⁻¹. As observed for Ca, the Mg content in the wines may be related to its soil content (Table 1), filtering agents, content of alcohol, and tartrates and sulfates in the wines (GARCIA, 1988).

The P content for the wines from the FI treatment were higher than the RDI and DI treatments, with mean values of 97.67 mg L⁻¹, 85.67 mg L⁻¹, and 70.68 mg L⁻¹, respectively. The minerals present in the wines are extracted mainly from the

skin of the grape during the maceration process (RIZZON; SALVADOR; MIELE, 2008). Conditions promoting an increase in soil moisture, as in FI treatment, make elements carried by diffusive flow, such as P (COSTA et al., 2006), more available for the plants and therefore contributing to a higher uptake of that element by grapevines. Phosphorus is found in wine usually from 50 mg L⁻¹ to 120 mg L⁻¹, and the content found in this study was within the range established for all the evaluated treatments. Its presence in wines promotes stronger aroma and flavor characteristics and, at high content, the formation of ferric phosphate precipitation occurs, known as ferric casse, causing turbidity (RIBÉREAU-GAYON et al., 2003).

The K content was similar in all the wines from the three treatments evaluated, and was between 1789.43 mg L⁻¹ (RDI) and 1804.07 mg L⁻¹ (DI), which is in agreement with the results found by Pereira et al. (2007), between 1835.9 mg L⁻¹

¹ and 3671.8 mg L⁻¹, in different red wines from the Northeast of Brazil. In contrast, the K content found was above those described in the literature, of 400 mg L⁻¹ to 1500 mg L⁻¹, in temperate climate conditions and soil from the South of Brazil (RIZZON; SALVADOR; MIELE, 2008). It is possible that the soils from the semi-arid region have a higher K content and therefore contribute to a higher content of this element in the wine. The high K content in wine may cause the formation and precipitation of potassium bitartrate. Therefore, the quantification of this element in wine is important to avoid precipitation which can be achieved through techniques such as stabilization by cold treatment, or by the use of ion exchange resins (PEYNAUD, 1997). Despite the high content of this mineral found in this study, its precipitation in wines was not observed. In addition, the high K values may be related to the high pH values, which may cause microbiological instability, fast evolution, and degradation of wines (PEYNAUD, 1997).

The Na content in wines is associated with the use of enological products in winemaking as well as its geographic origin, which means, the location where the grape was produced and the wine was elaborated (RIZZON; SALVADOR; MIELE, 2008). Brazilian legislation states that the maximum Na content is 200 mg L⁻¹, whereas Rizzon (2010) stated that the contents described in the literature are between 5 mg L⁻¹ and 50 mg L⁻¹ for the South of Brazil, which corroborates the results obtained in this work. The values determined in the present work were 26.06 mg L⁻¹ for the RDI treatment, 8.23 mg L⁻¹ for the FI treatment, and 15.64 mg L⁻¹ for the DI treatment (Table 3).

The Cu content of the wines from the FI treatment were higher than those from RDI treatment, with mean values of

0.32 mg L⁻¹ and 0.13 mg L⁻¹, respectively. Oliveira et al. (2015) also observed higher Cu contents in the berries from FI treatment and attributed this result to the greater availability of water which favored the transport and, consequently, uptake of Cu by the grapevine. Among the micronutrients, Cu is one of the least mobile in the soil due to its strong adsorption on the organic and inorganic colloids of the soil, and diffusion is one of the main means of transport of this element (SILVA; MENDONÇA, 2007).

The Cu from the must may come from the phytosanitary treatments of the grapevine or through the contact of the must with materials that contain Cu (RIZZON; SALVADOR; MIELE, 2008). Thus, independently of the irrigation treatments evaluated, the Cu contents found in the present study are associated with the Cu-based fungicides used in the control of mildew in the vineyard (Forum®). The maximum Cu content allowed in wines is 1.0 mg L⁻¹ (RIZZON; SALVADOR; MIELE, 2008). All the samples evaluated from the treatments were within the limits established by the literature and according to limits found in Pereira et al. (2007), of 0.1 mg L⁻¹ to 0.3 mg L⁻¹ in wines from the Northeast of Brazil.

Iron is a cation found in all wines, and when present in high content, due oxidation processes, there is precipitate formation altering the turbidity in the wines. The content in wine was similar among the evaluated treatments, with means of 0.72 mg L⁻¹ to 0.88 mg L⁻¹. The Fe content usually varies from 3 mg L⁻¹ to 7 mg L⁻¹ (RIZZON; SALVADOR; MIELE, 2008), which is above the content found in this study, and that can be explained by lower Fe contents in the soil, according to Oliveira et al. (2015).

The Mn content of the wine from the RDI treatment group was higher than that from the FI and DI treatments, with

means of 2.12 mg L^{-1} , 1.79 mg L^{-1} , and 1.84 mg L^{-1} , respectively, which are within the published limits, varying from 0.5 mg L^{-1} to 3.5 mg L^{-1} (DUTRA et al., 2011).

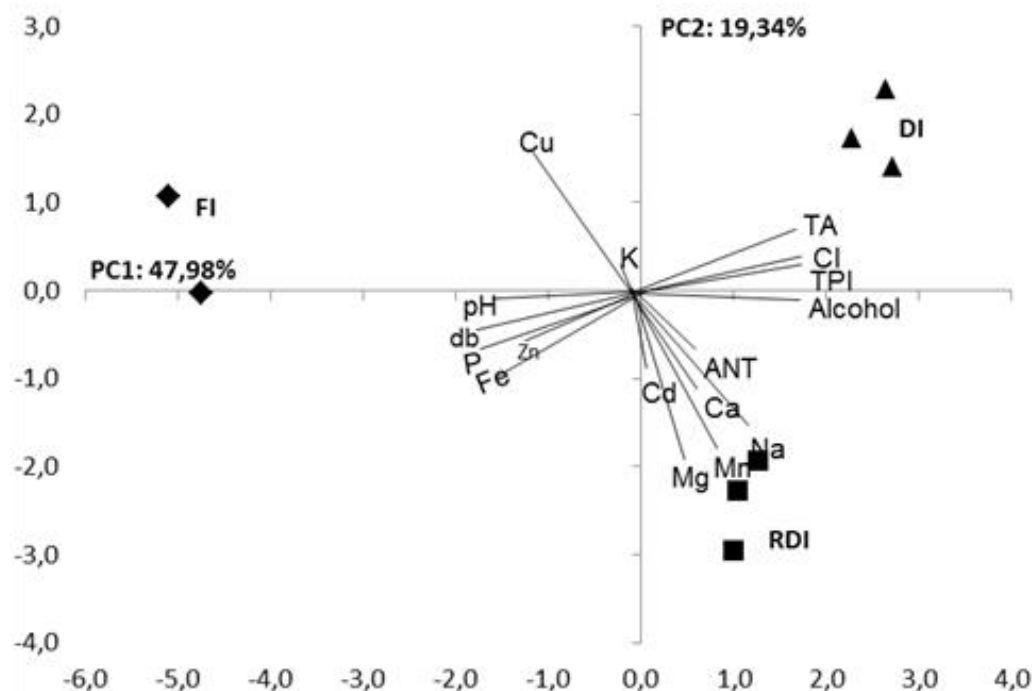
Zinc was found at a very low level in wines, and an eventual increase may be a consequence of contact with certain galvanized materials or certain alloys made/containing this metal (PEYNAUD, 1997). The Zn content remained between 0.59 and 0.61 mg L^{-1} for the treatments evaluated.

The presence of heavy metals in wine is directly associated with the development of industrial activity and the soil and water contamination. However, according to Ribéreau-Gayon et al. (1998), these elements are naturally present in the must and wines in a non-toxic level (endogenous presence). The Cd content remained between 0.04 mg L^{-1} and 0.05 mg L^{-1} for all the treatments evaluated, and is in agreement with the mean levels cited by Dutra et al. (2011). The Cr content was on average 0.01 mg L^{-1} (Table 4). According to the literature, the Cr content is usually present at content lower than 0.06 mg L^{-1} in the wines (RIBÉREAU-GAYON et al., 1998).

5.4 Principal Component Analysis (PCA)

According to Figure 1, PCA applied to all results, including those of the wine's physicochemical and minerals analyzes, allowed to a very good discrimination of the three irrigation treatments. The principal component 1 (PC1) and PC2 explained 67.32% of the total variability. The samples are represented by vertices, and each one represents a replicate performed and the proximity between them represents good reproducibility among the evaluations. On the other hand, the physicochemical and mineral variables evaluated were represented as vectors, and the resulting vectors explain the segmentation of samples with regard to the axes. The higher the result from a given vector in a given axis, the greater is the importance of the attribute to segment the samples on that axis. Thus, the PCA is important to graphically illustrate the similarity or difference among groups of individuals or treatments, to explain the variation between individuals, and to identify the sources and/or causes of variations. In the present study, physicochemical parameters and the minerals present in the wines, elaborated from three treatments of irrigation strategies, were used to carry out the multivariate statistical analysis. The PC1 explained 47.98%, whereas the PC2 explained 19.34% of the total variability.

Figure 1. Principal component analysis (PCA) of the physicochemical properties and mineral composition of wines elaborated from the full irrigation (FI), regulated deficit irrigation (RDI) and deficit irrigation (DI) treatments.



db = density, alcohol = alcohol content, TA = total acidity, CI = color intensity, ANT = anthocyanins, TPI = total polyphenols index, Cu = copper, P = phosphorus, K = potassium, Ca = calcium, Mg = magnesium, Mn = manganese, Na = sodium, Zn = zinc, Fe = iron, Cd = cadmium, Cr = chromium (Cr).

The PCA separated the wines into three distinct groups according to their physicochemical properties and mineral composition. The first group consisted of the wines elaborated with grapes subjected to the FI treatment and it is located in the negative part of PC1; these wines were characterized by their pH, density, P, Fe, Cu and K, where the values of P and Cu were the higher ones for this treatment. The pH, density, K, and Fe were not significantly different in the treatments evaluated (Tables 2 and 3). However, these were important for the PCA, presenting vectors with higher values. The second group was formed by the wines from the DI treatment, located in the positive part of the x-axis (PC1), which were characterized by total acidity, color intensity, TPI, and alcohol content, and where the greater means for these parameters were found in this treatment (Table 2). The third group

was represented by the wines from the RDI treatment, characterized by Mg, Na, Ca, Cd, and by total anthocyanins. All these results presented by the PCA can be confirmed in Tables 2 and 3.

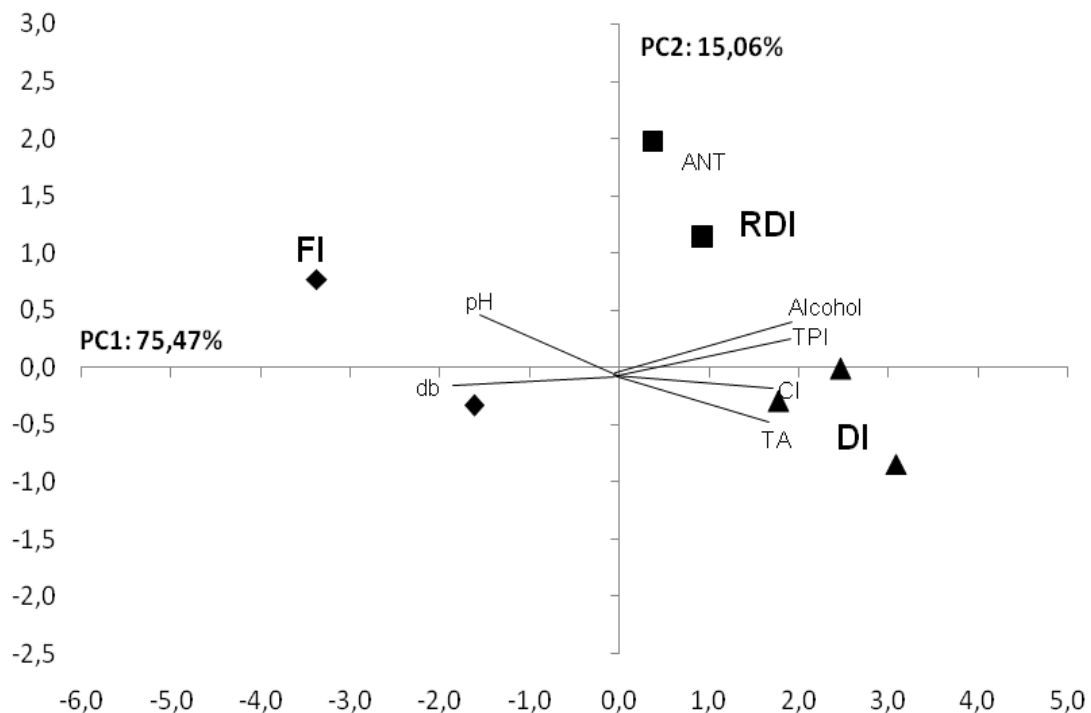
The PCA was also applied to the physicochemical and mineral composition parameters in separated statistical models. In both cases, we observed the separation of treatments, with different factors explaining the models.

Figure 2 shows the graphic of PCA only for the physicochemical variables of the wines studied. The x and y-axes (PC1 and PC2) explained 90.53% of total variability. The PC1 was the one that better represented this variation, showing 75.47%, whereas PC2 explained 15.06%. For the FI group, located in the negative part of the PC1, the variables that represent these wines were the pH and relative density (Table 2). The variables that

characterize the wines from the RDI treatment, located in the positive part of the x and y axes, were the total anthocyanins, alcohol content, and TPI, whereas for the wines from the DI treatment, located in the positive part of x

axis and negative part of y axis, the critical parameters were color index, and total acidity. These data can be verified in Table 2, where the means for the physicochemical properties of wines are shown.

Figure 2. Principal component analysis (PCA) of the physicochemical properties of wines elaborated from the full irrigation (FI), regulated deficit irrigation (RDI) and deficit irrigation (DI) treatments.

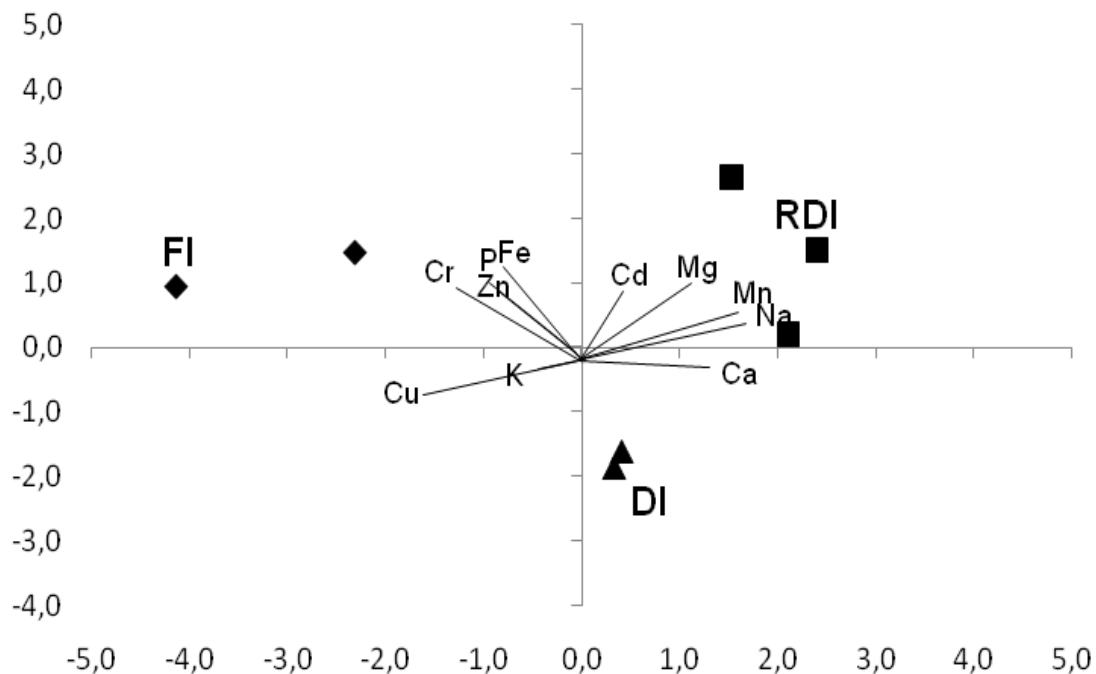


dB = density, alcohol = alcohol content, TA = total acidity, CI = color intensity, ANT = anthocyanins, TPI = total polyphenols index.

The x and y axes (PC1 and PC2) obtained with the principal component analysis (PCA) of the mineral characterization of wines elaborated from grapevines subjected to three different irrigation strategies (FI, RDI and DI) explained 66.92% of total variability (Figure 3). The PC1 explained 40.32%, whereas the PC2 explained 26.60%. The minerals that characterized wines from FI treatment, located in the negative part of the PC1, were Fe, P, Zn and Cr, Cu and K. Regarding the wines from the RDI treatment, located in the positive parts of PC1 and PC2, Mn, Mg, Na, and Cd

characterized them. The wines from the DI treatment, located in the positive parts of PC1 and negative part of the PC2, were inversely characterized with the minerals Fe, Cr, Zn, and P. All these results shown for PCA can be seen in Table 3. Although not showing significant differences by the Tukey's test (Table 3), the elements Fe, Cr and Zn representing the wines from the FI treatment, the Cd representing the wines from the RDI treatment, and the Ca representing those from the DI treatment, were significant in the multivariate analysis by the PCA.

Figure 3. Principal component analysis (PCA) obtained from the mineral composition of wines elaborated from the full irrigation (FI), regulated deficit irrigation (RDI) and deficit irrigation (DI) treatments.



Cu = copper, P = phosphorus, K = potassium, Ca = calcium, Mg = magnesium, Mn = manganese, Na = sodium, Zn = zinc, Fe = iron, Cd = cadmium, Cr = chrome.

When comparing Figures 1, 2 and 3, we could verify that the results from the physicochemical properties were more sensitive to variations in the irrigation strategies when compared to the analyses of the mineral composition. This is because the percentage of variables explained in the PC1 was 47.98% for all the variables together, 75.47% for the physicochemical properties, and 40.32% for the mineral composition. Therefore, the analyses of physicochemical properties were more efficient in discriminating the treatments evaluated than the analyses of mineral contents.

The present study showed that wines obtained from grapevines subjected to different irrigation strategies presented different properties and enological potentials. There is no best wine from three irrigation treatments, but there are different wines with variable physicochemical

properties, indicated for several kinds of consumers, according to taste and price. This suggests that, by crossing the analytical characteristics, the quantities of water applied to each treatment, and the costs of the different levels of water, a specific management for the elaboration of specific wines can be identified. For example, full irrigation, in which water is supplied from the pruning until harvest and consequently uses more water and energy for pumping, should present a higher production cost, high grape production and the winemaking of cheaper young wines with low tannic structure. On the other hand, the water deficit treatments (RDI and DI) apply less water, use less energy for pumping, and produce more concentrated grapes, which allow the winemaking of greater structure and phenolic content wines, in lower quantities but with higher added value. Therefore, depending on the

market niche, a specific irrigation strategy can be chosen to obtain commercial wines that should please a higher number of consumers, with different requirements in terms of wine typicality and price.

6 CONCLUSIONS

The results of the univariate analyses indicated that different irrigation strategies influenced the physicochemical properties of the wines, except for the anthocyanins. Multivariate statistical analysis, by using principal component analysis (PCA), showed that the total anthocyanins were important to characterize wines from the regulated deficit irrigation (RDI).

The RDI and deficit irrigation (DI) favored higher alcohol content, TPI, and color intensity. RDI also favored higher Mg, Na, and Mn contents, whereas the P

was higher in the full irrigation (FI). The Cd, Cr, Fe and Zn were not influenced by the irrigation strategies.

The wines demonstrated different physicochemical properties and mineral composition with different wine typicalities. The irrigation strategies influenced the physicochemical properties of wines in the Lower Middle São Francisco Valley. This enabled the production of wines with specific characteristics that should please many consumers with different tastes.

7 ACKNOWLEDGMENTS

The authors acknowledge the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for the financial support, and the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) for the fellowship.

8 REFERENCES

BASSOI, L. H.; GONÇALVES, S. O.; SANTOS, A. R. L.; SILVA, J. A.; LIMA, A. C. M. Influência de manejos de irrigação sobre aspectos de ecofisiologia e de produção da videira cv. Syrah/Paulsen 1103. *Irriga*, Botucatu, v. 16, p. 395-402, 2011.

BASSOI, L. H.; DANTAS, B. F.; LIMA FILHO, J. M. P.; LIMA, M. A. C.; LEÃO, P. C. S.; SILVA, D. J.; MAIA, J. L. T.; SOUZA, C. R.; SILVA, J. A. M.; RAMOS, M. M. Preliminary results of a long-term experiment about RDI and PRD irrigation strategies in winegrapes in São Francisco Valley, Brazil. *Acta Horticulturae*, The Hague, v. 754, p. 275-282, 2007.

BRASIL. Decreto N° 8.198, de 20 de Fevereiro de 2014. Regulamenta a Lei n° 7.678, de 8 de novembro de 1988, que dispõe sobre a produção, circulação e comercialização do vinho e derivados da uva e do vinho. *Diário Oficial da União*, Brasília, DF, 21 fev. 2014. Edição Extra 1.

BRASIL. Lei N° 10.970, de 12 de novembro de 2004. Altera dispositivos da Lei n° 7.678, de 8 de novembro de 1988, que dispõe sobre a produção, circulação e comercialização do vinho e derivados da uva e do vinho, e dá outras providências. *Diário Oficial da União*, Brasília, DF, 16 nov. 2004. Seção 1, p. 1.

- COSTA, J. P. V.; BARROS, N. F.; ALBUQUERQUE, A. W.; MOURA FILHO; G.; SANTOS, J. R. Fluxo difusivo de fósforo em função de doses e da umidade do solo. **Revista Brasileira de Engenharia Agrícola e Ambiental**, Campina Grande, v. 10, p. 828-835, 2006.
- DONAGEMA, G. K.; CAMPOS, D. B.; CALDERANO, S. B.; TEIXEIRA, W. G.; VIANA, J. M. **Manual de métodos de análise de solos**. 2. ed. Rio de Janeiro: Embrapa Solos, 2011. 230 p.
- DUTRA, S. V.; ADAMI, L.; MARCON, A. R.; CARNIELI, G. J.; ROANI, C. A.; SPINELLI, F. R.; LEONARDELLI, S.; DUCATTI, C.; MOREIRA, M. Z.; VANDERLINDE, R. Determination of the geographical origin of Brazilian wines by isotope and mineral analysis. **Analytical and Bioanalytical Chemistry**, Heidelberg, v. 401, p. 1571-1576, 2011.
- ETCHEBARNE, F.; OJEDA, H.; DELOIRE, A. Grape berry mineral composition in relation to vine water status and leaf area/fruit ratio. In: ROUBELAKIS-ANGELAKIS, K. A. **Grapevine Molecular Physiology & Biotechnology**. Berlin: Springer, 2009. p. 53-72.
- GARCIA, A. S. C. **Controlo de qualidade dos vinhos: química enológica: métodos analíticos**. Lisboa: Instituto da Vinha e do Vinho, 1988. 420 p.
- GARCÍA-ESTÉVEZ, I.; ANDRÉS-GARCÍA, P.; ALCALDE-EON, C.; GIACOSA, S.; ROLLE, L.; RIVAS-GONZALO, J. C.; QUIJADA-MORÍN, N.; ESCRIBANO-BAILÓN, M. T. Relationship between agronomic parameters, phenolic composition of grape skin, and texture properties of *Vitis vinifera* L. cv. Tempranillo. **Journal of Agricultural and Food Chemistry**, Washington, v. 63, p. 7663-7669, 2015.
- NICULCEA, M.; MARTINEZ-LAPUENTE, L.; GUADALUPE, Z.; SÁNCHEZ-DÍAZ, M.; AYESTARÁN, B.; ANTOLÍN, M. C. Characterization of phenolic composition of *Vitis vinifera* L. 'Tempranillo' and 'Graciano' subjected to deficit irrigation during berry development. **Vitis**, Siebeldingen, v. 54, p. 9-16, 2015.
- OFFICE INTERNATIONAL DE LA VIGNE ET DU VIN. **Compendium of international methods of analysis of vines and musts**. Paris: OIV, 2016. v. 1, 504 p.
- OLIVEIRA, V. S.; LIMA, A. M. N.; SALVIANO, A. M.; BASSOI, L. H.; PEREIRA, G. E. Heavy metals and micronutrients in the soil and grapevine under different irrigation strategies. **Revista Brasileira de Ciência do Solo**, Viçosa, v. 39 p. 62-173, 2015.

- OLLÉ, D.; GUIRAUD, J. L.; SOUQUET, J. M.; TERRIER, N.; AGEORGES, A.; CHEYNIER, V.; VERRIES, C. Effect of pre- and post-veraison water deficit on proanthocyanidin and anthocyanin accumulation during Shiraz berry development. **Australian Journal of Grape and Wine Research**, Glen Osmond, v. 17, p. 90-100, 2011.
- PADILHA, C. V. S.; BIASOTO, A. C. T.; CORREA, L. C.; LIMA, M. S.; PEREIRA, G. E. Phenolic compounds and antioxidant activity of commercial tropical red wines (*Vitis vinifera* L.) from São Francisco valley, Brazil. **Journal of Food Biochemistry**, Malden, v. 41, p. 1-9, 2017.
- PEREIRA, G. E.; SOARES, J. M.; GUERRA, C. C.; LIRA, M. M. P.; LIMA, M. D. O.; SANTOS, J. O. Caractérisation de vins rouges tropicaux produits au Nord-Est du Brésil. In: DEUTSCHER WEINBAUKONGRESS, 59.; INTERNATIONALES SYMPOSIUM INNOVATIONEN DER KELLERWIRTSCHAFT, 8., 2007, Stuttgart. **Anais...** Stuttgart: OIV, 2007. p. 1-4.
- PEYNAUD, E. **Connaissance et Travail du Vin**. Paris: Dunod, 1997. 341 p.
- REYNIER, A. **Manuel de viticulture**. Paris: Tec & Doc Editions, 2007. 532 p.
- RIBÉREAU-GAYON, P. R.; DUBOURDIEU, D.; DONÈCHE, B.; LONVAUD, A. **Tratado de enología**: microbiologia del vino vinificaciones. Buenos Aires: Editorial Hemisfério Sul, 2003. 654 p.
- RIBÉREAU-GAYON, P.; GLORIES, Y.; MAUJEAN, A.; DUBOURDIEU, D. **Traité d'Oenologie**: chimie du vin: stabilisation et traitements. Paris: Dunod, 1998. 519 p.
- RIZZON, L. A. Análises clássicas dos vinhos. In: RIZZON, L. A. (Ed.). **Metodologia para análise de vinho**. Brasília, DF: Embrapa Informação Tecnológica; Bento Gonçalves: Embrapa Uva e Vinho, 2010. p. 13-55.
- RIZZON, L. A.; SALVADOR, M. B. G.; MIELE, A. Teores de cátions dos vinhos da Serra Gaúcha. **Ciência e Tecnologia de Alimentos**, Campinas, v. 28, p. 635-641, 2008.
- ROMERO, P.; GARCÍA, J. G.; FERNÁNDEZ-FERNÁNDEZ, J. I.; MUÑOZ, R. G.; SAAVEDRA, F. A.; MARTÍNEZ-CUTILLAS, A. Improving berry and wine quality attributes and vineyard economic efficiency by long-term deficit irrigation practices under semiarid conditions. **Scientia Horticulturae**, Amsterdam, v. 203, p. 69-85, 2016.
- SANTESTEBAN, L. G.; MIRANDA, C.; ROYO, J. B. Regulated deficit irrigation effects on growth, yield, grape quality and individual anthocyanin composition in *Vitis vinifera* L. cv. 'Tempranillo'. **Agricultural Water Management**, Amsterdam, v. 98, p. 1171-1179, 2011.
- SANTOS, H. G.; JACOMINE, P. K. T.; ANJOS, L. H. C.; OLIVEIRA, V. A.; LUMBRERAS, J. F.; COELHO, M. R.; ALMEIDA, J. A.; ARAUJO FILHO, J. C.; OLIVEIRA, J. B.; CUNHA, T. J. F. **Sistema Brasileiro de Classificação de Solos**. 5. ed. Brasília, DF: Embrapa, 2018. 356 p.

SILVA, I. R.; MENDONÇA, E. S. Matéria orgânica do solo. In: NOVAIS, R. F.; ALVAREZ, V. V. H.; FONTES, R. L. F.; CANTARUTTI, R. B.; NEVES, J. C. L. (Org.). **Fertilidade do solo**. Viçosa: Sociedade Brasileira de Ciência do Solo, 2007. p. 275-374.

SOIL SURVEY STAFF. **Keys to Soil Taxonomy**. 12th ed. Washington, D.C.: USDA - Natural Resources Conservation Service, 2014. 360 p.

USSEGLIO-TOMASSET, L. **Chimie oenologique**. Paris: Tec & Doc, 1995. 387 p.

VELEDA, D.; MONTAGNE, R.; ARAUJO, M.; PEREIRA, G. E.; TYAQUIÇÃ, P.; NORIEGA, C.; LACERDA, F. Tropical Atlantic variability impacts on the Sub-middle São Francisco Valley, a Brazilian wine-producing area. **Global Journal of Agricultural Research Reviews**, Haveri, v. 3, p. 133-145, 2015.

ZIÓŁKOWSKA, A.; WAŚOWICZ, E.; JELEŃ, H. H. Differentiation of wines according to grape variety and geographical origin based on volatiles profiling using SPME-MS and SPME-GC/MS methods. **Food Chemistry**, Barking, v. 213, p. 714-720, 2016.