OENO One vine & wine

DOI: https://doi.org/10.20870/oeno-one.2024.58.1.7799

OPEN ACCESS

ORIGINAL RESEARCH ARTICLE

Potassium fertigation and organic fertilisation in 'Syrah' grape in Northeastern Brazil: yield, must characteristics and phenolic compounds

Alexsandro Oliveira da Silva[1], Bruno Ricardo Silva Costa[2], Henrique Oldoni[2], Davi José Silva[3] and Luis Henrique Bassoi[4*]

[1] Universidade Federal do Ceará, Depto. de Engenharia Agrícola, Av. Mister Hull 2977, Bloco 804, 60455-760, Fortaleza, CE, Brasil
[2] FCA UNESP Avenida Universitária 3780, 18610-034, Botucatu, SP, Brasil
[3] Embrapa Semiárido, BR-428 km 152, 56302-970, Petrolina , PE, Brasil
[4] Embrapa Instrumentação, Rua XV de Novembro 1452, 13561-206, São Carlos, SP, Brasil

*correspondence: luis.bassoi@embrapa.br

Associate editor: Vittorino Novello

Received: 8 October 2023

Accepted: 16 December 2023

Published: 23 January 2024

ABSTRACT

The effect of both potassium and organic fertilisation on grape yield and must composition originating from wine grapevines cultivated in the São Francisco river basin, in the semi-arid region of Northeastern Brazil, is not well known. Since soils from that region usually have low organic matter content, we hypothesise that soil application of organic fertiliser and potassium application through drip irrigation enhances the availability of this nutrient in the soil, and it influences grape yield and quality. Hence, five doses of potassium (0, 20, 40, 80 and 160 kg ha^{-1}) applied through a drip irrigation system and two doses of goat manure as organic fertiliser applied into the soil (0 and 7.5 m^3 ha^{-1}) after pruning were evaluated concerning yield and berry quality of grapevine 'Syrah' over four growing seasons (April 2013 to July 2015) in Petrolina, state of Pernambuco, Brazil. The experimental design was randomised blocks with 5 replications. We evaluated yield (t ha^{-1}), weight of 100 berries (g), must volume (mL), total soluble solids (°Brix), pH and total titratable acidity (‰), K, Mg and Ca contents and phenolic compounds (total polyphenols, anthocyanins and flavonoids). The increasing application of potassium at the different doses of organic fertiliser did not alter the crop yield, except in the second season. Nonetheless, K concentration in grape must was influenced in the four growing seasons, with higher values at fitted K_2O doses by polynomial regression of 83.62 kg ha^{-1} (first season), 101.25 kg ha^{-1} (second season), 120.00 kg ha^{-1} (third season) and 77.99 kg ha^{-1} (0 m^3 ha^{-1} organic fertiliser) and 96.07 kg ha^{-1} (7.5 m^3 ha^{-1} organic fertiliser) in the fourth season. The addition of increasing doses of potassium interfered with the wine grape quality, which is an important factor to be considered in relation to its acidity. Organic fertilisation did not affect the most quality characteristics of the grapes.

KEYWORDS: drip irrigation, fertiliser management, grape quality, Brazilian semi-arid

INTRODUCTION

Potassium (K) is the most abundant cation in the grape berry and it plays a role in berry sugar accumulation, berry maturation, berry water relations, cellular growth, disease resistance, abiotic stress tolerance, mitigating senescence and in the flow of xylem and phloem vessels (Mpelasoka *et al.*, 2003; Brunetto *et al.*, 2015; Haddad and Kamangar, 2015; Keller *et al.*, 2015; Rogiers *et al.*, 2017). In addition, K significantly influences the pH and total titratable acidity of grape must, but it has little effect on the concentration of phenolic compounds, such as anthocyanins and flavonoids (Delgado *et al.*, 2004; Morris *et al.*, 1987).

Grape growing develops worldwide, even within semi-arid areas such as the Lower-middle region of the São Francisco River basin in the Brazilian Northeast. Particularly for this region, it was demonstrated that grape yield is minimally impacted by K fertilisation (Silva *et al.*, 2023). However, high levels of exchangeable potassium (K^+) in the soil can alter grape components, including total soluble solids and pH of the grape must (Ciotta *et al.*, 2016; van Leeuwen *et al.*, 2018) as well as to lead a magnesium deficiency in the grapevine (Ramos and Martínez de Toda, 2019).

Organic matter improves several soil attributes, but there are different sources of it for fertilisation purposes. In this context, due to the goat farming commonly performed in the semi-arid region of Brazil, goat manure is an alternative to be utilised as organic fertiliser (Rocha *et al.*, 2015; Silva *et al.*, 2016). This region is particularly deficient in soil organic matter due to its fast decomposition, induced by high temperatures (Assis *et al.*, 2010) and little input of organic material (Queiroz *et al.*, 2018). The interaction between organic matter and potassium fertiliser has not been studied for the purpose of wine grape production in that region.

The use of 30 m^3 ha^{-1} of goat manure increased the soil phosphorus content and the cation exchange capacity and also increased the yield of grapevines ‘Syrah’ grown under this semi-arid condition. Concurrently, a decrease in the total titratable acidity of grape must was observed (Rocha *et al.*, 2015). Increments in Ca, Mg, Mn and NO_3^- concentrations in the soil were also observed (Silva *et al.*, 2016) with goat manure fertilisation and nitrogen fertigation in ‘Syrah’ grape.

The effects of K and manure fertilisation on the yield of wine grapes grown in the Brazilian semi-arid and its influence on grape must composition are not well known. Since soils from that region usually have low organic matter content, as reported herein, we hypothesise that soil application of organic fertiliser and potassium application through drip irrigation enhances the availability of this nutrient in the soil, and it influences grape yield and quality. Thus, this study aimed to evaluate whether different levels of K and organic fertilisation affect the yield and must composition of wine grapes ‘Syrah’ cultivated in a semi-arid region.

MATERIALS AND METHODS

1. Description of the experimental area

The study was carried out in an experimental field of Brazilian Agricultural Research Corporation (Embrapa), in Petrolina, state of Pernambuco, Brazil (9° 8’ 8.9” S latitude, 40° 18’ 33.6” W longitude, 373 m altitude). We evaluated the grapevine ‘Syrah’ (*Vitis vinifera* L.) grafted onto Paulsen 1103 rootstock and planted in a 0.35 ha vineyard in April 2009, in a 1.0 × 3.0 m spacing between plants and rows, respectively. Plants were supported by a trellis system and grown with lateral branches with six spurs per plant and three buds per spur. The grapevines were irrigated by a drip irrigation system, with emitters spaced at 0.5 m and 2.5 L h^{-1} flow rate.

The gross irrigation depth (I, mm) supplied the water demand corresponding to the crop evapotranspiration (ETc, mm) (Table 1). ETc was calculated as a product of the reference evapotranspiration (ETo, mm) estimated by the Penman–Monteith method (Allen *et al.*, 2021) and the crop coefficient (kc) for each phenological stage of the ‘Syrah’ grapevines grown in this experimental area (Bassoi *et al.*, 2007).

TABLE 1. Reference evapotranspiration (ETo, mm), crop evapotranspiration (ETc, mm), precipitation (P, mm) and gross irrigation depth (I, mm) in four growing seasons of ‘Syrah’ grapevine.

Growing Season	dap	ETo	ETc	ETc-P	I
		mm			
1st	117	617.4	458.9	420.7	496.9
2nd	117	743.0	565.4	374.9	416.5
3rd	118	659.9	463.8	451.4	502.1
4th	121	547.4	523.8	343.3	381.0

dap - days after pruning.

The soil of the vineyard was categorised as a Typic Plinthustalf (Soil Survey Staff, 2014) and presented a loamy sand texture, with 81, 13 and 6 g kg^{-1} of sand, silt and clay, respectively. Table 2 presents the results of the soil chemical analysis performed in January 2013.

TABLE 2. Chemical analysis of the Typic Plinthustalf in the 0-0.20 m and 0.20-0.40 m layers.

Depth	pH $CaCl_2$	EC	OM	P	K	Ca	Mg	H+Al	Na	CEC	V
(m)		dSm^{-1}	g dm^{-3}	mg dm^{-3}			cmol$_c$ dm^{-3}				(%)
0-0.20	7.1	0.43	11.7	77.8	0.35	3.12	1.33	0.80	0.11	5.71	84
0.20-0.40	6.8	0.25	4.8	63.5	0.20	2.37	1.03	1.57	0.08	5.25	70

EC: electrical conductivity; OM: organic matter; K: potassium; Ca: calcium; Mg: magnesium; Na: sodium; H+Al: potential acidity; CEC: cation exchange capacity; V: base saturation.

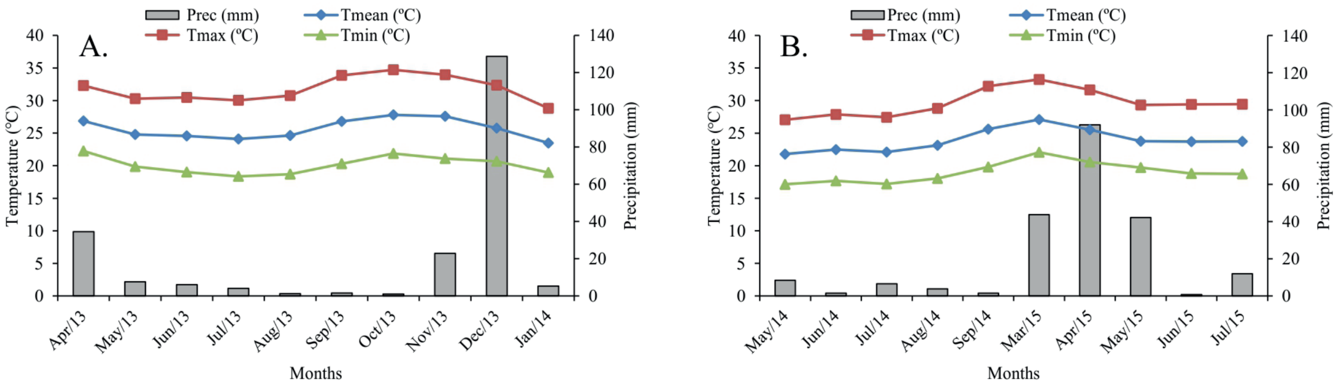

FIGURE 1. Precipitation (P) and air temperature (maximum: T_{max}; mean: T_{mean}; and minimum: T_{min}) data recorded during the first and second growing seasons (A) and third and fourth growing seasons (B) of 'Syrah' grape in Petrolina.

Chemical and physical analyses were performed as described by scientific laboratory methods (Teixeira *et al.*, 2017).

The study lasted for three years, comprising four consecutive growing seasons. Throughout all, weather data were recorded by an automatic station located 60 m away from the experimental field (Figure 1).

2. Experimental design and treatments

The treatments consisted of five doses of potassium oxide, K_2O, (0, 20, 40, 80 and 160 kg ha^{-1}) and two doses of goat manure (0 and 7.5 m^3 ha^{-1}), adopted as organic fertiliser (OF). They were arranged into a randomised complete block design with five replications, assuming a split-plot design. The K_2O and OF doses were assigned within the subplots and whole plots, respectively. The experimental unit (EU) comprised 16 plants per plot, and 8 plants per plot were considered for analysis.

The chemical analysis of the OF (Table 3) was performed according to scientific methodology (Silva, 2009). It was applied atop the soil prior to the spur pruning carried out on April 29, 2023, October 4, 2013, May 30, 2014 and March 9, 2015. These dates marked the beginning of the first, second, third and fourth growing seasons, respectively.

Potassium fertilisation was performed for 10 weeks, starting one week after the spur pruning, via fertigation, using an injector pump with a flow rate of 360 L h^{-1}. The potassium fertilisers used were potassium chloride (K_2O = 60 %), potassium nitrate (K_2O = 45 %) and potassium sulfate (K_2O = 50 %), and they were applied separately in the proportion of 50 %, 40 % and 10 % of the total K_2O dose applied, respectively. These fertilisers were used to achieve the desired amount of potassium to be applied by fertigation. The accompanying ions were balanced using complementary fertilisation.

3. Grape yield and qualitative and nutritional aspects of must

Harvests were carried out at 117 (first and second seasons), 118 (third season) and 121 (fourth season) days after pruning (dap), respectively, on August 23, 2013, January 29, 2014, September 26, 2014 and July 8, 2015. The grapes were weighted on a scale (0.01 g readability) to calculate the yield (t ha^{-1}) from the average weight of grapes per plant. The weight of 100 grape berries was determined on a scale with a 0.01 g accuracy; subsequently, the grapes were crushed to obtain the must and determine its volume (mL), total soluble solids (TSS) with portable digital refractometer (°Brix), pH with a digital benchtop pH meter and total titratable acidity (TTA) by titration with 0.1 N NaOH, using bromothymol blue as an indicator (Ciotta *et al.*, 2016).

Chemical analyses were performed to determine the mineral content (K, Ca and Mg) of the grape must. This analysis was conducted using the dry method in a muffle furnace at 500 °C, with K determined by flame photometry and Ca and Mg by atomic absorption spectrophotometry (Rizzon and Miele, 2012).

4. Phenolic compounds analysis

To determine the total extractable polyphenols (TEP), 1.0 g of peel and 5.5 g of pulp were weighed, then 50 % methanol and 70 % acetone were added (25 mL each), followed by centrifugation (15,000 rpm) for 15 minutes after each addition of reagent, and readings were performed with the extract. Folin–Ciocalteau and gallic acid were used as standard in the UV/VIS spectrophotometer at 280 nm wavelength.

TABLE 3. Chemical composition of goat manure used as organic fertiliser.

pH	EC	N	P	K	Ca	Mg	S	B	Cu	Fe	Mn	Zn	Na
	dS m^{-1}				——— g kg^{-1} ———						——— mg kg^{-1} ———		
5.1	0.32	3.5	2.2	13.8	13.1	4.7	2.3	37.3	21.2	4495.0	338.7	55.2	1482.7

EC: electrical conductivity; N: nitrogen; P: phosphorus; K: potassium; Ca: calcium; Mg: magnesium; S: sulfur; B: boron; Cu: cooper; Fe: iron; Mn: manganese; ZN: zinc; Na: sodium.

The calibration curve was used to determine the quantity of total polyphenols according to the absorbance reading obtained. Anthocyanins and flavonoids were determined by weighing 0.5 g of grape peel and adding a solution (25 mL) with ethanol and 1.5 N HCl; after 24-hour rest, the samples were filtered using hydrophilic cotton. Readings were performed with the extract in UV/VIS spectrophotometer at 535 and 374 nm wavelength.

5. Statistical analysis of results

The analysis of variance (ANOVA) was performed to account for differences between treatments, setting the significance level at 5 % ($p \leq 0.05$). Shapiro–Wilk's and Bartlett's tests were adopted to check the assumptions of normality of residuals and homoscedasticity of variances, respectively. The regression analysis was performed if a significant main effect of interaction ($K_2O \times OF$) was found ($p \leq 0.05$) or a single main effect of K_2O. Then, the relationship between K_2O doses and the variable of interest was characterised by linear or quadratic functions according to the significance of the regression coefficients. For a significant single main effect of OF, we performed the Student's t-test or the Welsh's t-test (if the groups do not have equal variances) to compare the means of the variable of interest. Additionally, the Principal Component Analysis (PCA) was applied to the technological and phenolic ripening and the mineral concentration in grape must obtained in each growing season. PCA was performed using PAleontological STatistics—PAST software version 4.02 (Natural History Museum/University of Oslo).

RESULTS

1. Grape yield and grape must quality

Potassium and organic fertilisation did not influence grape yield in three growing seasons, which was equal to 4.0 t ha^{-1} (0 m^3 ha^{-1} OF) and 4.2 t ha^{-1} (7.5 m^3 ha^{-1} OF) in the first season, 8.6 t ha^{-1} (0 m^3 ha^{-1} OF) and 8.2 t ha^{-1} (7.5 m^3 ha^{-1} OF) in the third season, and 2.6 t ha^{-1} (0 m^3 ha^{-1} OF) and 2.4 t ha^{-1} (7.5 m^3 ha^{-1} OF) in the fourth season.

K_2O application significantly interfered with must pH in the first season at the different OF doses used and in the third season for the single effect of K_2O doses. For the first season, the data were described by a quadratic regression model, with maximum fitted values of 80.00 and 114.28 kg ha^{-1} of K_2O for OF doses of 0 and 7.5 m³ ha^{-1}, respectively. For the third season, the fitted regression model was linear, with a 0.0006 decrease in pH for each unit increase in K_2O doses (Table 4).

For TTA (Table 5), the influence ($p \leq 0.01$) of K_2O doses was observed in all growing seasons, demonstrating the strong relationship of this variable with the increase in K_2O availability in the soil. Interaction between OF and K_2O was observed only in the first season. During the first growing season, in the presence of organic fertilisation (7.5 m^3 ha^{-1}), there was a linear effect of potassium fertigation on total titratable acidity (TTA), with an increase of 0.009 % per unit increase in the K_2O doses applied, with no fit for the OF dose of 0 m^3 ha^{-1}. In the second growing season, there was a quadratic polynomial effect corresponding to the single effect of potassium fertigation on TTA. The fitted K_2O dose of 103.3 kg ha^{-1} led to the highest value of TTA, 6.45 ‰. In the third growing season, there was a linear effect of the K_2O doses on TTA, with an increase of 0.00449 % per unit increment in the K_2O dose applied. In the fourth growing season, there was a quadratic polynomial fit with the highest value of TTA, 7.06‰, at the fitted K_2O dose of 84.5 kg ha^{-1}.

TSS, the weight of 100 berries and the volume of the must were not significantly affected ($p > 0.05$) by the factors evaluated in the experiment, except for TSS in the second season (Table 6), in which soluble solids showed a quadratic behaviour, with reduction of ºBrix according to the increase in K_2O doses.

TABLE 4. pH in 'Syrah' grapes as function of different doses of potassium (K_2O) and organic fertiliser (OF).

OF (m^3 ha^{-1})	K_2O (kg ha^{-1}) 0	20	40	80	160	CV%	Equation	R^2
				First growing season (pH)				
0	3.64	3.68	3.84	3.69	3.67	1.21	$y = -0.000015x^2 + 0.0024x + 3.662$	0.30*
7.5	3.65	3.67	3.77	3.80	3.80		$y = -0.000014x^2 + 0.0032x + 3.637$	0.91*
				Second growing season (pH)				
0	3.78	3.90	3.86	3.84	3.92	3.43	ns	ns
7.5	3.90	3.82	3.92	3.94	3.94		ns	ns
				Third growing season (pH)				
-	3.80	3.90	3.50	3.51	3.40	10.99	$y = -0.0006x + 3.592$	0.64*
				Fourth growing season (pH)				
0	3.90	4.00	4.00	3.90	4.00	5.05	ns	ns
7.5	3.80	4.00	4.00	3.80	4.00		ns	ns

ns = not significant * significant at 5 % probability of error.

TABLE 5. Total titratable acidity (TTA) in 'Syrah' grape as function of different doses of potassium (K_2O) and organic fertiliser (OF).

	OF (m^3 ha^{-1})	K_2O (kg ha^{-1}) 0	20	40	80	160	CV%	Equation	R^2
First growing season (TTA, ‰)	0	7.42	7.42	6.97	7.98	7.61	4.43	ns	
	7.5	7.24	7.16	7.31	7.39	7.91		$y = 0.0045^{**}x + 7.1325$	0.91*
Second growing season (TTA, ‰)	-	6.1	6.28	6.30	6.44	6.36	7.25	$y = -0.00003x^2 + 0.0062x + 6.1313$	0.94*
Third growing season (TTA, ‰)	-	5.71	5.83	6.46	6.06	6.63	11.39	$y = 0.0049x + 5.8463$	0.61*
Fourth growing season (TTA, ‰)	-	6.06	6.90	7.20	6.69	6.51	7.90	$y = -0.0001x^2 + 0.0169x + 6.3508$	0.46*

ns = not significant * significant at 5 % probability of error.

TABLE 6. Total soluble solids in must (TSS), volume of must (V) and mass of 100 berries (M) of 'Syrah' grape as function of different doses of potassium (K_2O) and organic fertiliser (OF).

OF (m^3 ha^{-1})	K_2O (kg ha^{-1}) 0	20	40	80	160	CV%	Equation	R^2
				First growing season				
				TSS (°Brix)				
0	22.8	22.4	22.9	22.3	22.2	3.16	ns	ns
7.5	22.8	23.1	22.6	22.8	22.3		ns	ns
				V (mL)				
0	83.2	84.7	88.9	88.5	82.5	5.03	ns	ns
7.5	84.0	84.0	86.0	81.2	84.8		ns	ns
				M (g)				
0	171	167	170	168	159	5.82	ns	ns
7.5	160	164	170	163	160		ns	ns
				Second growing season				
				TSS (°Brix)				
0	20.4	20.8	21.2	20.2	21.8	5.03	ns	ns
7.5	20.8	20.8	20.0	20.6	21.2		$y = 0.000137x^2 - 0.0199x + 23.895$	0.52*
				V (mL)				
0	57	58	64	59	58	6.13	ns	ns
7.5	57	59	64	66	61		ns	ns
				M (g)				
0	142	142	153	148	153	10.57	ns	ns
7.5	147	148	155	155	150		ns	ns
				Third growing season				
				TSS (°Brix)				
0	21.5	21.4	20.7	21.4	19.8	6.13	ns	ns
7.5	19.9	20.2	20.6	20.9	21.2		ns	ns
				V (mL)				
0	80	71	75	68	78	1.13	ns	ns
7.5	71	74	72	77	78		ns	ns
				M (g)				
0	163	152	159	151	166	8.55	ns	ns
7.5	162	155	165	164	163		ns	ns
				Fourth growing season				
				TSS (°Brix)				
0	20.4	20.8	21.2	20.2	21.8	5.03	ns	ns
7.5	20.8	20.8	20.0	20.6	21.2		ns	ns
				V (mL)				
0	92	96	103	108	113	0.96	ns	ns
7.5	95	100	102	102	116		ns	ns
				M (g)				
0	171	174	177	180	182	1.59	ns	ns
7.5	172	174	181	180	180		ns	ns

ns = not significant * significant at 5 % probability of error.

2. Nutrient and phenolic compounds content in the grape must

For K concentration in grape must (Table 7), significant effects were observed in all growing seasons. In the first season, there was a polynomial fit for the K_2O doses, and the highest K concentration (3384 mg L^{-1}) was observed at the fitted K_2O dose of 83.62 kg ha^{-1}. In the second season, there was a polynomial fit, with the lowest value of K (1953.2 mg L^{-1}) observed at the fitted K_2O dose of 101.25 kg ha^{-1}. In the third growing season, only the K_2O dose factor influenced the concentration of K in the must, and the fit was linear with an increase of 5.66 mg L^{-1} for each unit increase of K_2O. In the fourth season, there was an interaction between the studied factors (K_2O doses × OF doses) with the polynomial fit ($p \leq 0.05$) for the concentration of K in the grape must. For the OF dose of 0 m^3 ha^{-1}, there was a decrease in the concentration of K as a function of the K_2O doses, with the lowest value (1038 mg L^{-1}) reaching the fitted K_2O dose of 77.99 kg ha^{-1}. For the OF dose of 7.5 m^3 ha^{-1}, there was an increasing polynomial fit as a function of the K_2O doses up to the adjusted value of 96.07 kg ha^{-1}, with an accumulation of 3295 mg L^{-1} of K in the must.

The concentration of Ca in the must was not influenced ($p > 0.05$) by the treatments in any of the growing seasons, showing mean values of 27.02 (first season), 17.32 (second season), 37.66 (third season) and 124.12 mg L^{-1} (fourth growing seasons).

The concentration of Mg in the must (Table 8) was influenced by the interaction of treatments (K_2O and OF doses) in the first growing season and by the single factor K_2O doses in the fourth growing season. For the first season, the fit was quadratic, with the highest values (39.37 and 42.01 mg L^{-1} at OF doses of 0 and 7.5 m^3 ha^{-1}, respectively) observed at fitted K_2O doses of 57.17 kg ha^{-1} (0 m^3 ha^{-1}) and 72.85 kg ha^{-1} (7.5 m^3 ha^{-1}). In the fourth growing season, Mg concentration was influenced by the K_2O dose factor, with the quadratic polynomial fit and the highest value (30.61 mg L^{-1}) at the fitted K_2O dose of 120.00 kg ha^{-1}.

K_2O and OF application did not significantly affect the phenolic compounds, except in the first growing season for anthocyanins ($p \leq 0.05$), assuming the interaction between K_2O and OF doses and in the third growing season for flavonoids in response to K_2O doses ($p \leq 0.01$) as a single factor. For the concentration of anthocyanins (Table 9), for

TABLE 7. Potassium concentration in 'Syrah' grape as function of different doses of potassium (K_2O) and organic fertiliser (OF).

OF	K_2O (kg ha^{-1})						Equation	R^2
(m^3 ha^{-1})	0	20	40	80	160	CV%		
	First growing season (K, mg L^{-1})							
-	2592	2852	3004	3504	2640	21.18	$y = -0.1239x^2+20.72x+2517.8$	0.92*
	Second growing season (K, mg L^{-1})							
-	3290	2768	2128	2232	2296	24.38	$y = 0.1057x^2-21.405x+3036.9$	0.84*
	Third growing season (K, mg L^{-1})							
-	1052	1100	960	1064	1956	15.22	$y = 5.66x + 886.8$	0.75*
	Fourth growing season (K, mg L^{-1})							
0	2512	1592	1200	1224	2448	30.7	$y = 0.2149x^2-33.522x+2345.5$	0.70*
7.5	1000	1424	1440	3976	1952		$y = -0.2969x^2+57.052x+554.34$	0.93*

ns = not significant * significant at 5 % probability of error.

TABLE 8. Magnesium concentration in 'Syrah' grape as function of different doses of potassium (K_2O) and organic fertiliser (OF).

OF (m^3 ha^{-1})	K_2O (kg ha^{-1})						Equation	R^2
	0	20	40	80	160	CV%		
	First growing season (Mg, mg L^{-1})							
0	34.6	36.0	37.2	39.4	19.0	8.85	$y = -0.0018x^2+0.2058x+33.489$	0.97*
7.5	19.0	39.8	39.0	38.2	17.6		$y = -0.0034x^2+0.4954x+23.972$	0.82*
	Second growing season (Mg, mg L^{-1})							
0	42.7	40.8	41.5	41.7	41.7	3.95	ns	ns
7.5	41.4	40.9	41.5	39.9	41.3		ns	ns
	Third growing season (Mg, mg L^{-1})							
-	28.1	27.6	26.9	28.7	30.7	5.67	$y = 0.0196x+27.26$	0.62*
	Fourth growing season (Mg, mg L^{-1})							
-	29.9	28.4	27.4	27.3	30.6	9.28	$y = 0.0005x^2-0.0735x+29.82$	0.98*

ns = not significant * significant at 5 % probability of error.

TABLE 9. Concentration of anthocyanins and flavonoids in 'Syrah' grape as function of different doses of potassium (K_2O) and organic fertiliser (OF).

OF (m^3 ha^{-1})	K_2O (kg ha^{-1}) 0	20	40	80	160	CV%	Equation	R^2
	First growing season—Anthocyanins (mg 100 g^{-1})							
0	614.2	548.2	644.6	525.2	657.2	21.75	ns	
7.5	581.4	653.6	901.8	749.6	642.6		$y = -0.0334x^2+5.519x+601.52$	0.57*
	Third growing season—Flavonoids (mg 100 g^{-1})							
-	182.6	200.6	179.8	171.7	159.0	15.37	$y = -0.203x+190.99$	0.70*

ns = not significant * significant at 5 % probability of error.

the OF dose of 7.5 m^3 ha^{-1}, a quadratic fit was observed with maximum values at the K_2O dose of 82.61 kg ha^{-1}. For the concentration of flavonoids, the fit was linear, with a decrease of 0.203 mg 100 g^{-1} for each unit increase in K_2O doses.

3. Organic fertiliser effect

We also observed the single main effect of organic fertilisation on certain 'Syrah' wine grapes traits at harvest. Significant differences were found between the means of yield ($p \leq 0.01$) and must volume ($p \leq 0.01$) at the second growing season. Therefore, the Student's t-test showed that the goat manure fertilisation promoted a decrease in yield (Figure 2A) but an increase in must volume (Figure 2B). The flavonoid content was only affected by the organic fertilisation in the third growing season ($p \leq 0.01$). According to the Student's t-test, the mean flavonoid content in grape must of unfertilised plants was higher than in fertilised plants (Figure 2C). The mineral content in grape must was also influenced by the organic fertilisation alone and we noticed this single main effect on Ca concentration in the second ($p \leq 0.01$) and third growing season ($p \leq 0.05$) and on Mg concentration in the fourth growing season ($p \leq 0.01$). Thus, the Student's t-test showed that the mean Ca concentration was higher in grape must of unfertilised plants than in fertilised plants (Figures 2D and 2E). Nevertheless, Welsh's t-test indicated that the organic fertilisation resulted in an improvement in Mg mean concentration (Figure 2F).

4. Principal Component Analysis (PCA)

Based on the results of the PCA, the first two principal components (PC1 and PC2) were considered in each of the growing seasons (Figure 3), which accounted for more than 55 % of the explanation of the total variance. For the first growing season (Figure 3A), 55.14 % of the data variance was explained by the first two components, of which 34.67 % was explained by PC1 and 20.47 % was explained by PC2.

The variables K, flavonoids, anthocyanins, TSS, TTA, pH showed a high contribution in the variation of PC1, while the nutrients Ca and Mg (highly) and the total polyphenols (TEP) contributed to the variation of PC2, leading to the formation of two groups: the first formed by the K_2O doses of 20 kg ha^{-1} (7.5 m^3 ha^{-1}), 40 kg ha^{-1} (0 and 7.5 m^3 ha^{-1}) and 80 kg ha^{-1} (7.5 m^3 ha^{-1}) with the greatest influence on the variables pH, TSS, K, flavonoids and anthocyanins; and the second by the K_2O doses of 0 and 80 kg ha^{-1}, with greater influence of 0 m^3 ha^{-1} on the variables Ca, TTA and TEP. In addition, the nutrients Ca and Mg were negatively correlated with treatments with the highest applied dose of K_2O (160 kg ha^{-1}), regardless of the organic fertiliser; that is, when grape plants were fertilised with high levels of K_2O, there was a reduction of these nutrients (Ca and Mg) in the berry.

In the second season (Figure 3B), 61.89 % of the total variance of the data was explained by the first two components together, 34.84 % by PC1 and 27.05 % by PC2. The variables TEP, pH, TSS, Ca and Mg had a greater contribution to PC1, while anthocyanins, flavonoids, TTA and K had a greater contribution to PC2. An important highlight was observed in this season regarding the OF factor, differing from that observed in the univariate analysis, where distinct groups of data without (0 m^3 ha^{-1}) and with (7.5 m^3 ha^{-1}) organic fertiliser were identified, regardless of the levels of K_2O, separated mainly by the axis of PC2. In addition, treatments with higher levels of K_2O (40 and 80 kg ha^{-1} of K_2O applied) and with OF were associated with higher TTA values. The addition of OF and K_2O in general, especially at the highest doses, increased the pH of the must.

In the third season (Figure 3C), the first two principal components explained 63.79 % of the total variance of the data, 39.33 % for PC1 and 24.46 % for PC2. The variables TEP, anthocyanins, TTA, K and Mg had a greater contribution to PC1, while the variables pH, soluble solids and Ca had a higher contribution to PC2. As in the second season, but in a more evident way, there was separation of groups in relation to the use of OF; the presence of OF promoted an increase in pH and reduction of flavonoids, TSS and Ca in the must at most doses studied. The addition of 160 kg of K_2O ha^{-1} increased TTA and the concentrations of K and Mg but led to musts with lower concentrations of anthocyanins and TEP. These variables were found at higher concentrations in grape musts from treatments with lower doses of K_2O and in which OF was not used.

For the fourth season (Figure 3D), the total variance of the data, explained by the first two components, was 58.8 %, with PC1 and PC2 explaining 33.42 % and 25.38 %, respectively. The application of 80 and 160 K_2O ha^{-1} together with organic fertiliser increased K concentrations in the must and, on the other hand, reduced the levels of TEP, TTA and pH, which were negatively correlated with the concentration of K in the must.

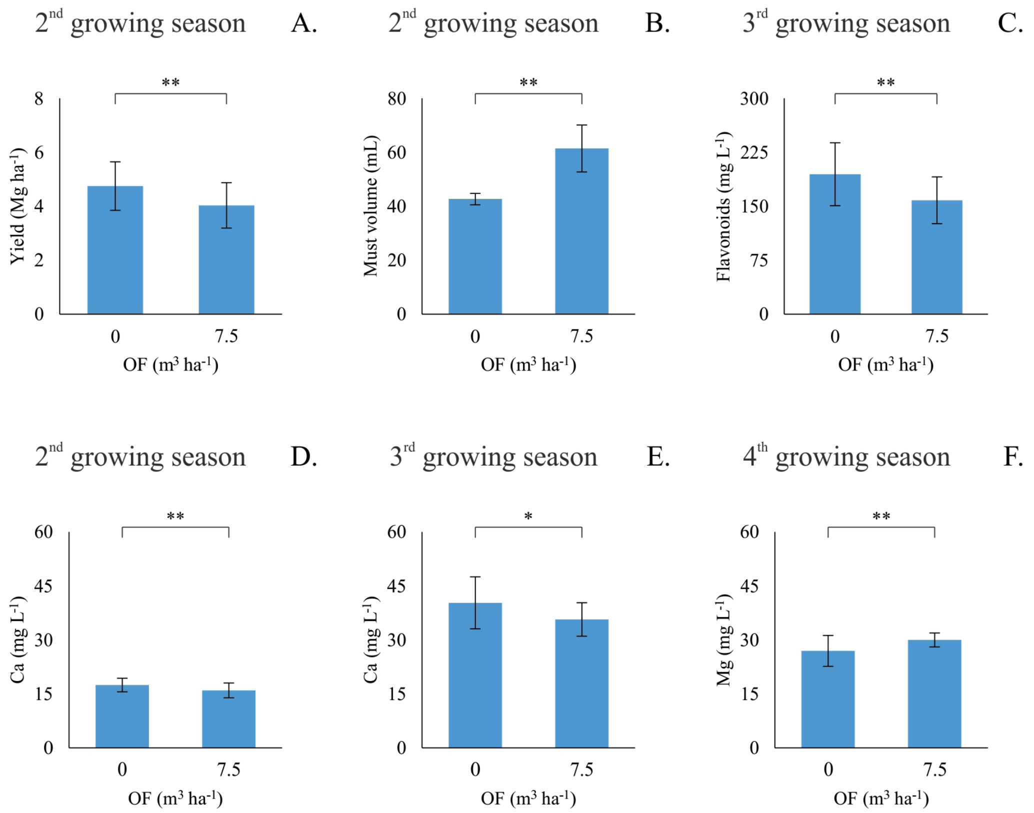

FIGURE 2. Mean and standard deviation bars of wine grape traits for each organic fertiliser rate. * and ** indicate a difference at the 0.05 and 0.01 significance level according to Student's t test or Welsh's t test.

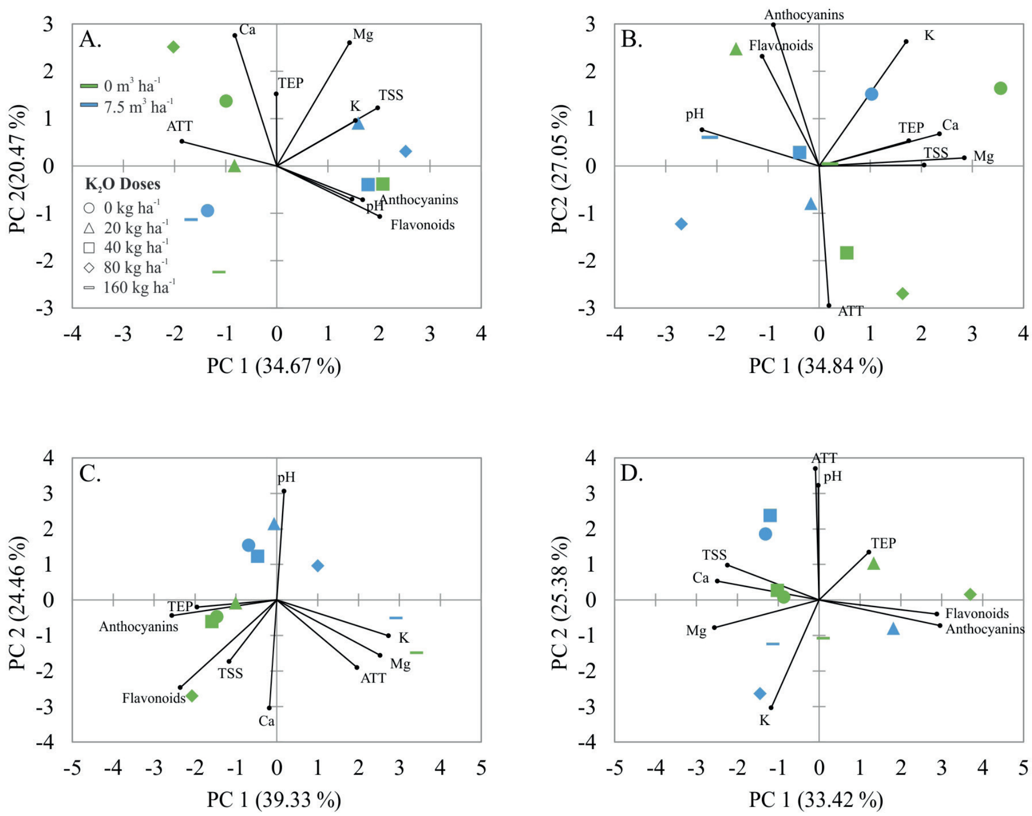

FIGURE 3. Principal component analysis for quality variables of the must from grapevines subjected to different doses of potassium and organic fertiliser in Petrolina (A, B, C and D correspond to first, second, third and fourth growing seasons, respectively).

DISCUSSION

Excess potassium caused significant differences in the pH of Cabernet-Sauvignon grapes (Morris *et al.*, 1987), as observed in this study in the first and third growing seasons (Table 4), while no effect of K on the pH of Cabernet-Sauvignon grape must was observed (Poni *et al.*, 2003) as reported herein for second and fourth seasons (Table 4). As 50 % of the total K absorbed is accumulated in the berries, the chemical composition of the must, by excess K, can alter other components such as total titratable acidity (Delgado *et al.*, 2004; Morris *et al.*, 1987; van Leeuwen *et al.*, 2018), without influencing crop yield (Silva *et al.*, 2023) mainly in soils with high concentrations of K. The increase in soil potassium caused an increase of 0.047 in must TTA in 'Cynthiana' wine grapes due to the increase in total soluble solids (Morris *et al.*, 1987). The same behaviour has been observed (Karimi, 2017), i.e., an increase in TTA values due to the addition of K_2O. Such information shows that the increase in TTA caused by the increment of K_2O in the soil in the present experiment (Table 5) results from the addition of potassium, being observed in the four growing seasons. TSS showed no differences related to potassium fertilisation (Morris *et al.*, 1987) as observed in the first, second, third (without OF) and fourth seasons, while the only response to potassium doses was together OF in the third season (Table 6).

The increase of K concentration in grapes with the addition of K_2O in the soil (Table 7) is corroborated by other studies with 'Cabernet-Sauvignon' (Poni *et al.*, 2003) and for 'Zweigelt' (Griesser *et al.*, 2017), where the berries contain one of the highest concentrations of K among the parts of the plant. Another study (Howell and Conradie, 2013) reported a higher concentration of K in the composition of the must (2600 mg L^{-1}), with frequent applications of K_2O via fertigation (91.9 kg ha^{-1}). More than 50 % of the total K absorbed by plants is accumulated in the berries, and its functions are enzymatic activation and synthesis of reactions, contributing to fruit maturation (Brunetto *et al.*, 2015). Positive correlations between the soil extractable K and the foliar K, the K in the must and the K in the wines were reported by Martínez-Vidaurre *et al.* (2023).

Although Ca is an essential element for the structure of grape berries, being responsible for the rigidity of their cell wall (Brunetto *et al.*, 2015), no response of grape Ca concentration was found for both treatments. On the other hand, there was a response in Mg concentration in grapes and the variation of K concentration in the plants in the first, second and fourth seasons (Table 8). Higher Mg values in the juice of grapes produced with a K application of 91.9 kg ha^{-1} were reported (Howell and Conradie, 2013) as well as the variation of K^+ in the plants altered the concentration of Mg in the grapes (Griesser *et al.*, 2017). The increase in Mg concentration improves chlorophyll synthesis in the plant, but the increase in concentration in grape berries is possibly related to their maturation and the high absorption of K in this process (Brunetto *et al.*, 2015).

Differences in the values of anthocyanins and flavonoids were found only, respectively, in the first and third seasons (Table 9). The values of anthocyanin and phenolic compounds observed in this experiment were higher than in other studies (e.g., Bassoi *et al.*, 2007) with different irrigation managements for the same variety and under the same edaphoclimatic conditions. Possibly, potassium fertilisation has a greater influence on the phenolic compounds evaluated than water deficit does, thus increasing their concentration in the grape berries.

No differences were observed in relation to yield and must volume in three growing seasons, but observed an evolution of different elements in the soil as organic matter, phosphorus content and cation exchange complex (Rocha *et al.*, 2015). Increasing Ca, Mg, Mn and NO^{3-} in the same region were reported in a previous study carried out over three growing seasons from April 2010 to September 2011 with goat manure and nitrogen fertigation (Silva *et al.*, 2016). OF applied in this experiment was probably not sufficient to change yield, must volume, flavonoids, calcium and magnesium content (Figure 2). Especially with respect to the maintenance of organic matter content in the soil, soil cultivation has caused significant losses in the irrigated Brazilian semi-arid area, being more expressive in annual cropping. It is highly recommended that cropping systems consider more effective cultivation control strategies, such as crop rotation, favouring the addition of plant residues to the soil for effective soil organic matter recycling (Assis *et al.*, 2010). The amount of organic matter in the soil of Brazilian semi-arid zones is usually low, and the use of management systems that aim their accumulation and maintenance at appropriate levels will contribute to the increase in the CEC, as well as to improve their chemical and physical properties (Queiroz *et al.*, 2018).

In general, the Ca content was negatively correlated with the values of pH; on the other hand, it was positively correlated with the levels of anthocyanins and flavonoids in most seasons. These effects may be related to the addition of K_2O in the soil, which is a determinant for increasing the values of most parameters evaluated (Walker and Blackmore, 2012). The relationships between the variables evaluated herein have been observed in several other studies involving potassium fertilisation (Ciotta *et al.*, 2016; Walker and Blackmore, 2012) and water deficit (Vila Nova *et al.*, 2019) in grapes, despite the differences in climate and soil fertility of the different sites.

Harvests were performed in August 2013, January and September 2014 and July 2015. Possibly, seasonality influenced the variability of the studied variables, which was observed in the correlation between Ca and TEP and the concentration of K in the must. A study demonstrated the effect of seasonality in the São Francisco Valley region, where phenolic components of wine, such as flavonoids, ranged from 1150.0 to 2142.2 mg L^{-1} between 2014 and 2016 for 'Syrah' grapes (Oliveira *et al.*, 2019). In another study carried out in the same region (Oliveira *et al.*, 2020), the intra-annual climate variability influenced the composition of grapes and wines as grape harvesting occurs throughout the year.

Higher concentrations for most of the phenolic indices evaluated (total phenols, flavonoids, total and coloured anthocyanins, total pigments, condensed tannins and proanthocyanidins), as well as for colour intensity, alcohol strength and total dry extract in the second semester of the year (warmest season).

CONCLUSIONS

The addition of increasing doses of potassium through drip irrigation in a Typic Plinthustalf soil cultivated with 'Syrah' grapevines throughout four growing seasons in the Brazilian semiarid region interfered with the grape quality, being an important factor to be considered in relation to its acidity. Moreover, this work has provided new information related to the joint effect of potassium and organic fertilisation for wine vine growing in that region. Despite the low organic content in the soil, goat manure application did not affect the most quality characteristics of the grape must evaluated in this study.

ACKNOWLEDGEMENTS

To Coordenação de Pessoal de Nível Superior (CAPES), for the fellowship to the first author.

REFERENCES

Allen, R.G., Dhungel, R., Dhungana, B., Huntington, J., Kilic, A. & Morton, C. (2021). Conditioning point and gridded weather data under aridity conditions for calculation of reference evapotranspiration. *Agricultural Water Management, 245*(1),1-21. https://doi.org/10.1016/j.agwat.2020.106531

Assis, C. P., Oliveira, T. S., Dantas, J. A. N. & Mendonça, E. S. (2010). Organic matter and phosphorus fractions in irrigated agroecosystems in a semi-arid region of Northeastern Brazil. *Agriculture, Ecosystems and Environment*, *138*, 74-82. https://doi.org/10.1016/j.agee.2010.04.002

Bassoi, L.H., Dantas, B.F., Lima Filho, J. M. P., Lima, M. A. C., Leão, P. C. S., Silva, D. J., Maia, J. T. L. & Souza C. R. (2007). Preliminary results of a long-term experiment about RDI and PRD irrigation strategies in winegrapes in São Francisco Valley, Brazil. *Acta Horticulturae, 754*, 275-282. https://doi.org/10.17660/ActaHortic.2007.754.35

Brunetto, G., Melo, G. W. B., Toselli, M., Quartieri, M. & Tagliavini, M. (2015). The role of mineral nutrition on yields and fruit quality in grapevine, pear and apple. *Revista Brasileira de Fruticultura, 37*(4), 1089-1104. https://doi.org/10.1590/0100-2945-103/15

Ciotta, M. N., Ceretta, C. A., Silva, L. O. S., Ferreira, P. A. A., Sautter, C. K., Couto, R. R. & Brunetto G. (2016). Grape yield, and must compounds of 'Cabernet-Sauvignon' grapevine in sandy soil with potassium contents increasing. *Ciência Rural, 46*(08), 1376-1383. https://doi.org/10.1590/0103-8478cr20150472

Delgado, R., Martín, P., Alamo, M. & González, M. R. (2004). Changes in the phenolic composition of grape berries during ripening in relation to vineyard nitrogen and potassium fertilisation. *Journal of the Science of Food and Agriculture, 84*, 623-630. https://doi.org/doi.org/10.1002/jsfa.1685

Griesser, M., Martinez, S. C., Weidinger, M. L., Kandler, W., & Forneck, A. (2017). Challenging the potassium deficiency hypothesis for induction of the ripening disorder berry shrivel in grapevine. *Scientia Horticulturae, 216*, 141-147. https://doi.org/10.1016/j.scienta.2016.12.030

Haddad, R. & Kamangar, A. (2015). The ameliorative effect of silicon and potassium on drought stressed grape (*Vitis vinifera* L.) leaves. *Iranian Journal of Genetics and Plant Breeding, 4*, 48-58.

Howell, C. L. & Conradie, W. J. (2013). Comparison of three different fertigation strategies for drip irrigated table grapes – part II. Soil and grapevine nutrient status. *South African Enology and Viticulture, 34*, 10-20. https://doi.org/10.21548/34-1-1024

Karimi, R. (2017). Potassium-induced freezing tolerance is associated with endogenous abscisic acid, polyamines and soluble sugars change grapevines. *Scientia Horticulturae, 215*, 184-194. https://doi.org/10.1016/j.scienta.2016.12.018

Keller, M., Zhang, Y., Shrestha, P. M., Biondi, M. & Bondada, B. R. (2015). Sugar demand of ripening grape berries leads to recycling of surplus phloem water via the xylem. *Plant, Cell and Environment, 38*, 1048-1059. https://doi.org/10.1111/pce.12465

Martínez-Vidaurre, J. M.; Pérez-Álvarez, E. P., García-Escudero, E. & Peregrina, F. (2023). Effects of soil water-holding capacity and soil $N\text{-}NO_3^-$ and K on the nutrient content, vigour and yield of cv. Tempranillo vine and the composition of its must and wine. *OENO One, 57*,2, 447-466. https://doi.org/10.20870/oeno-one.2023.57.2.7168

Morris, J. R., Simis, C. A., Striegler, R. K., Cackler, S. D. & Donley, R. A. (1987). Effects of cultivar, maturity, cluster thinning and expressing potassium fertilization on yield and quality of Arkansas wine grapes. *American Journal Enology and Vitiviniculture, 38*, 260-264. https://doi.org/10.5344/ajev.1987.38.4.260

Mpelasoka, B. S., Schachtman, D. P., Treeby, M. T. & Thomas, M. R. (2003). A review of potassium nutrition in grapevines with special emphasis on berry accumulation. *Australian Journal of Grape and Wine Research*, *9*,3, 154-168. https://doi.org/10.1111/j.1755-0238.2003.tb00265.x

Oliveira, J. B., Egipto, R., Laureano, O., Castro, R., Pereira, G. E. & Ricardo-da-Silva, J. M. (2019). Chemical composition and sensory profile of Syrah wine from semiarid tropical Brazil – rootstock and harvest season effects. *LWT – Food Science and Technology, 114*, 1-9. https://doi.org/10.1016/j.lwt.2019.108415

Oliveira, J. B., Laureano, O., Castro, R., Pereira, G. E. & Silva, J. M. R. (2020). Rootstock and harvest season affect the chemical composition and sensory analysis of grapes and wines of the Alicante Bouschet (*Vitis vinifera* L.) grown in a tropical semi-arid climate in Brazil. *OENO One, 4*, 1021-1039. https://doi.org/10.20870/oeno-one.2020.54.4.2553

Poni, S., Quartieri, M. & Tagliavini, M. (2003). Potassium nutrition of Cabernet Sauvignon grapevines (*Vitis vinifera* L.) as affected by shoot trimming. *Plant and soil, 253*, 341-351. https://doi.org/10.1023/A:1024832113098

Queiroz, A. F., Salviano, A. M., da Cunha, T. J. F., Olszevski, N., Souza Junior, V. S. & de Oliveira Neto, M. B. (2018). Potentialities and limitations of agricultural use in soils of semi-arid region of the state of Bahia. *Anais da Academia Brasileira de Ciências, 90*(4), 3373-3387. https://doi.org/10.1590/0001-3765201820180029

Ramos, M. C. & Martínez de Toda, F. (2019) Variability of Tempranillo grape composition in the Rioja DOCa (Spain) related to soil and climatic characteristics. *Journal of the Science of Food and Agriculture*, 99(3), 1153-1165. https://doi.org/10.1002/jsfa.9283

Rizzon, L. A. & Miele, A. (2012). Analytical characteristics and discrimination of Brazilian commercial grape juice, nectar, and beverage. *Ciência e Tecnologia de Alimentos, 32*(1), 93-97. https://doi.org/10.1590/S0101-20612012005000015

Rocha, M. G., Bassoi, L. H. & Silva, D. J. (2015). Soil attributes, irrigated 'Syrah' vine yield and must composition as function of organic and nitrogen fertilization. *Revista Brasileira de Fruticultura, 37*(1), 220-229. https://doi.org/10.1590/0100-2945-412/13

Rogiers, S. Y., Coetzee, Z. A., Walker, R. R., Deloire, A. & Tyerman, S. D. (2017). Potassium in the Grape (*Vitis vinifera* L.) Berry: Transport and Function. *Frontier in Plant Science, 8*, 1-19. https://doi.org/10.3389/fpls.2017.01629

Silva, A. O., Silva, D. J., Bassoi, L. H. & Chaves, A. R. M. (2023). NO^{-3}, K^{+}, and chlorophyll index in fertigated grapevines in the semi-arid region of Brazil. *Scientia Agricola, 80*, e20210122. https://doi.org/10.1590/1678-992X-2021-0122

Silva, D. J., Bassoi, L. H., Rocha, M. G., Silva, A. O. & Deon, M. D. (2016). Organic and Nitrogen Fertilization of Soil under 'Syrah' Grapevine: Effects on Soil Chemical Properties and Nitrate Concentration. *Revista Brasileira de Ciência do Solo, 40*, 1-11. https://doi.org/10.1590/18069657rbcs20150073

Silva, F. C. (2009). *Manual de análises químicas de solos, plantas e fertilizantes*. 2 ed Brasília: Embrapa Solos.

Soil Survey Staff (2014). *Keys to Soil Taxonomy*, 12 ed Washington: USDA-Natural Resources Conservation Service.

Teixeira, P. C., Donagemma, G. K., Fontana, A. & Teixeira, W. G. (2017). *Manual de métodos de análise de solo*. 3 ed Brasília: Embrapa Solos.

van Leeuwen, C., Roby, J. P. & Rességuier, L. (2018). Soil-related terroir factors: a review. *Oeno One, 52*(2), 173-188. https://doi.org/10.20870/oeno-one.2018.52.2.2208

Vila Nova, M., Rodríguez-Nogales, J. M., Vila-Crespo, J. & Yuste, J. (2019). Influence of water regime on yield components, must, composition and wine volatile compounds of Vitis vinifera cv. Verdejo. *Australian Journal of Grape and Wine Research, 25*, 83-91. https://doi.org/doi.org/10.1111/ajgw.12370

Walker, R. R. & Blackmore, D. H. (2012). Potassium concentration and pH inter-relationships in grape juice and wine of Chardonay and Shiraz from a range of rootstocks in different environments. *Australian Journal of Grape and Wine Research*, *18*, 183-193.