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Chemical characterization and mineral levels in the fruits of blackberry cultivars grown in a tropical climate at an elevation

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ABSTRACT. Sensorial attributes such as color, texture, acidity and nutritional composition are essential quality components for blackberry (*Rubus* spp.). The mineral content and quality of fruits of different blackberry cultivars produced in Lavras, Southern Minas Gerais, in a tropical climate at an elevation (Cwb, according to Köppen) were analyzed. The analyzed minerals were phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), zinc (Zn), manganese (Mn), copper (Cu) and iron (Fe). Additionally, chemical characteristics, the total acidity (citric acid), pH, soluble solids (°Brix) and vitamin C (ascorbic acid) level, and sensory characteristics, color (L*a*b) and firmness (N) were also evaluated. According to the results, the Choctaw and Xavante blackberry cultivars demonstrated the highest mineral contents, the Caingangue cultivar showed the highest soluble solid content, and the Ebano cultivar exhibited the highest vitamin C concentration and firmer fruits. The chemical variables in the fruits of the different blackberry cultivars presented little significant correlation with the contents of macrominerals present, whereas the opposite was obtained for the micronutrients. However, the chemical characteristics combined with the nutritional characteristics can be proposed as an excellent tool for the selection of cultivars of superior quality and high nutritional value.

Keywords: *Rubus* spp., micronutrients, macronutrients, vitamin C.

Caracterização química e teores de minerais em frutos de cultivares de amoreira-preta cultivadas em clima tropical de altitude

RESUMO. Os atributos sensoriais como cor, textura, acidez e a composição nutricional são componentes essenciais na qualidade da amora-preta (*Rubus* spp.). Qualidade e teores de minerais foram analisados em frutos de diferentes cultivares de amoreira-preta produzidas em Lavras, sul de Minas Gerais, clima tropical de altitude (Cwb, segundo Köppen). Os minerais analisados foram: fósforo (P), potássio (K), cálcio (Ca), magnésio (Mg), zinco (Zn), manganês (Mn), cobre (Cu) e ferro (Fe), bem como as seguintes características químicas: acidez total (ácido cítrico), pH, sólidos solúveis (Brix) e vitamina C (ácido ascórbico) e características sensoriais: cor (L*a*b*) e firmeza (N). De acordo com os resultados obtidos, as cultivares de amoreira-preta Choctaw e Xavante se destacaram com os maiores teores de minerais, a cultivar Caingangue maior teor de sólidos solúveis e a cultivar Ébano com maior concentração de vitamina C e frutos mais firmes. Verificou-se também que as variáveis químicas em frutos das diferentes cultivares de amoreira-preta apresentaram pouca correlação significativa com os teores de macrominerais presentes e o oposto foi obtido para os micronutrientes. Contudo, as caracteristicas químicas combinados com as características nutricionais podem ser propostos como excelentes ferramentas para seleção de cultivares de qualidade superior e elevado valor nutricional.

Palavras-chave: *Rubus* spp., micronutrientes, macronutrientes, vitamina C.

Introduction

Interest in the consumption of blackberries (*Rubus* spp.) has increased in recent years because the fruits possess high amounts of phenolic compounds and vitamin C, which can help prevent degenerative diseases (ALI et al., 2011; TIWARI et al., 2009). In addition to these compounds, the natural pigments, mainly anthocyanins, provide attractive colorants for the manufacture of dairy products, jellies and fruit syrup (ACOSTA-MONTOYA et al., 2010).

Approximately 400 cultivars of blackberry have been generated through genetic improvement efforts (CLARK; FINN, 2011), and their characteristics with regard to climatic adaptation to the various cultivation areas and the physical and chemical properties of their fruits are divergent (CAMPAGNOLO; PIO, 2012). Variations can even

occur in the contents of the chemical compounds in response to the cultivation locale due to the differences in the intensity of solar radiation and the temperature ranges that influence the organoleptic characteristics of the fruits (ALI et al., 2011; SIRIWOHARN et al., 2004).

The color of the fruit is initially of fundamental importance for consumer acceptance, followed by the firmness of the texture and the flavor. The quality attributes can be largely influenced by the availability of nutrients, which, together with sources of biologically active substances, vitamins and secondary metabolites, are necessary to ensure complete nutrition (CALLAHAN, 2003; CARL, 1999).

Among the cultivars, it is evident that various genetic factors exist that limit the quality and acceptance of the consumers, mainly in relation to the chemical composition of the fruits (SCALZO et al., 2005). However, the blackberry fruits cultivated in Brazil still need to be investigated in terms of characteristics that serve as fruit quality parameters, mainly for the regions that possess mild winters and summers with high temperatures.

Accordingly, the objective of the present work was to conduct a chemical and mineral content characterization of the fruits of blackberry cultivars cultivated under a tropical climate at an elevation.

Material and methods

The blackberry cultivars were sampled in the orchard of the Federal University of Lavras (UFLA), Lavras, Minas Gerais State. The municipal district is located at latitude 21°14'06" south and longitude 45°00'00" west, at an average elevation of 918 m. The climate of the area is the Cwb type, tropical altitude, according to the Köppen classification, and is characterized by dry winters and rainy summers and mild temperatures.

The fruits of ten blackberry cultivars (Arapaho, Brazos, Caingangue, Cherokee, Choctaw, Comanche, Ebano, Guarani, Tupy and Xavante) were randomly collected manually from various positions and orientations on the plants at the commercial maturation stage. The fruits were transported to the Biochemistry laboratory, Department of Chemistry at UFLA, where they were selected for the arrangement of the experimental units.

The experimental design was completely randomized, with four repetitions. The analyzed variables included the total titratable acidity (TTA), determined through a volumetric and potentiometric titration with a solution of NaOH 0.1N to the ideal pH range (8.2-8.4) (INSTITUTO ADOLFO LUTZ, 1985); the results were expressed as the % of the citric acid per 100 g^{-1} of fresh fruit. The pH of the fruits was analyzed using a pH meter (MB-10 São Paulo) Association of Official Analytical Chemists (AOAC, 2005). The vitamin C concentration (ascorbic acid mg per 100 g of fresh fruit) was determined according to Strohecker and Henning (1967). The coloration indexes of the fruits, brightness (L) varying from white (L = 100) to black (L = 0) and a*, characterizing coloration in the red (+a*) to the green (- a*) region and b* indicating coloration from the yellow (+b*) to the blue (- b*) range, were determined using a colorimeter (Chroma Meter model CR – 3000). For the firmness, the fruits were measured individually under the same conditions by determining the strength of resistance to compression under the condition of returning to the beginning using a texturometer (model TA-XT2 Stable Micro Systems Ltd., Godalming, UK) and the following operational parameters: probe, P $2N^{-1}$; perforation speed, 2 mm s^{-1} and pre-test speed, 1.5 mm s^{-1}. The perforation distance was standardized at 5 mm, and the results obtained were expressed in Newton (N).

The macromineral (P, K, Ca and Mg) and micromineral (Cu, Mn, Zn and Fe) contents were determined according to Malavolta et al. (1997). The extracts were obtained by nitropercloric digestion. The phosphorus and the sulfur levels were determined using colorimetry, according to the Association of Official Analytical Chemists (AOAC, 2005). The iron, zinc, manganese, copper, magnesium and calcium levels were determined using atomic absorption spectrophotometry and potassium using flame photometry. The results were expressed in milligrams per 100 g of fresh fruit, (mg 100 g^{-1}).

The data were analyzed using the Sisvar program (FERREIRA, 2011). The Pearson correlation coefficients were evaluated and calculated to 1% and 5% probabilities using the Student t test to correlate the quality attributes and the mineral elements measured in the blackberry fruits.

Results and discussion

The results of the mineral composition of the blackberry cultivar fruits are provided in Table 1. Potassium (K) was the most abundant mineral in the fruits of the cultivars, with an overall average of 129.80 mg 100 g^{-1}. The potassium content found for the Choctaw cultivar was 185.5 mg 100 g^{-1}, which was superior to that observed for the other cultivars.

The Choctaw, Tupy and Xavante cultivars presented the highest phosphorus (P) contents, and the lowest content was observed in the Ebano cultivar (Table 1). The highest calcium (Ca) content was found in the Cherokee and Xavante cultivars, with 23.3 and 22.1 mg 100 g^{-1}, respectively. Differences were also observed among the cultivars in relation to the magnesium (Mg) content, with a variation from 10.07 mg 100 g^{-1} (Ebano) to 21.40 mg 100 g^{-1} (Choctaw) and the average content being 15.95 mg 100 g^{-1} (Table 1).

The copper (Cu), manganese (Mn), zinc (Zn) and iron (Fe) micronutrient contents also varied among the blackberry cultivars, with averages of 0.10, 0.87, 0.23 and 2.55 mg 100 g^{-1}, respectively (Table 1).

The mineral contents in fruits are very dependent on the soil, fertilization, climatic conditions and cultivar (NOUR et al., 2011). As the management and manure application were the same for all of the cultivars, following the recommendations of Gonçalves et al. (2011), it is believed that such differences in the mineral contents are related to the characteristics that are intrinsic to each cultivar. These results are, therefore, important in the choice of a cultivar with superior properties for consumption, and even for facilitating genetic improvements to obtain superior cultivars. Significant variations were observed for the content of soluble solids, the pH and the acidity among the blackberry cultivars (Table 2). The Cherokee cultivar presented the highest content of soluble solids, followed by the Comanche, Tupy and Xavante cultivars, whereas the Ebano cultivar presented the lowest content (Table 2). Fruits with higher soluble solid values are the sweetest and more favored by the consumer market.

As the cultivars Brazos and Ebano had the lowest pH values, being more acidic, they are the most suitable for industry applications. Conversely, the fruits of the Choctaw cultivar had higher pH values, characterizing a less acid fruit, which is preferred for fresh fruit consumption (Table 2).

The quantitative characterization related to the color and texture is also indispensable for the producer in the choice of the use of the fruits. The highest firmness values, in other words, firmer fruits, were presented by the Ebano cultivar, followed by Cherokee. The lowest value, softer fruits, was observed for Tupy, followed by the Guarani cultivar (Table 2).

Because they are delicate, blackberry and raspberry (*Rubus idaeus*) fruits have low durability during postharvest storage (KRÜGER et al., 2011). As such, the evaluation of firmness is an important characteristic to be considered for the shelf-life of the fruits consumed fresh.

Table 1. Mean values of macronutrients phosphorus, potassium, calcium, magnesium and sulfur and micronutrients manganese, iron and zinc in the fresh matter of ten blackberry cultivars.

Cultivar	Mineral (mg 100 g^{-1})							
	P	K	Ca	Mg	Cu	Mn	Zn	Fe
Arapaho	17.1 b	143.3 b	20.2 b	17.4 b	0.09 d	0.71 e	0.18 e	1.38 e
Brazos	17.8 b	123.9 c	14.3 d	16.3 c	0.11 c	1.01 d	0.25 b	3.66 b
Caingangue	15.4 c	109.9 c	18.4 b	14.3 d	0.09 d	0.65 f	0.23 c	1.35 e
Cherokee	17.1 b	134.1 c	22.1 a	14.8 d	0.11 c	1.17 b	0.27 b	4.70 a
Choctaw	19.9 a	185.5 a	15.7 c	21.4 a	0.13 b	0.42 i	0.31 a	3.16 c
Comanche	15.7 c	117.0 c	16.2 c	15.7 c	0.11 c	0.59 g	0.24 c	3.62 b
Ebano	10.9 d	95.0 c	13.4 d	10.7 e	0.05 e	0.46 h	0.16 f	1.02 e
Guarani	15.6 a	116.8 c	16.8 c	15.8 c	0.10 d	1.17 b	0.26 b	1.71 e
Tupy	19.9 a	125.0 c	11.6 e	15.0 d	0.17 a	1.10 c	0.26 b	1.32 e
Xavante	19.6 a	147.1 b	23.3 a	17.7 b	0.09 d	1.47 a	0.20 d	2.47 d
Mean	16.94	129.80	16.26	15.95	0.10	0.87	0.23	2.44
CV%	4.78	11.72	6.96	4.92	7.13	3.29	4.70	11.16

Means followed by the same letter in a column belong to the same group according to Scott-Knott at a 5% probability ($p \leq 0.01$).

Table 2. Average color index values (L, a★ and b★), firmness (N), total titratable acidity (TTA - % citric acid), total soluble solids (TSS, expressed in °Brix), pH and vitamin C (AA - mg ascorbic acid 100 g^{-1}) in the fruits of ten blackberry cultivars.

Cultivar	Characteristic							
	L	a★	b★	N	TTA	TSS	pH	AA
Arapaho	18.86 a	8.08 d	-1.97 a	0.48 d	2.26 a	6.12 d	2.91 c	47.39 d
Brazos	17.22 b	10.26 b	-2.66 b	0.42 f	1.74 e	7.00 c	2.88 d	42.69 e
Caingangue	17.25 b	7.57 d	-3.69 c	0.63 c	0.68 h	7.25 c	2.95 c	50.36 c
Cherokee	18.44 a	9.23 c	-2.52 b	0.70 b	2.16 a	7.95 a	2.91 c	43.84 e
Choctaw	19.06 a	11.25 a	-1.32 a	0.41 f	1.91 c	7.00 c	3.13 a	46.10 d
Comanche	17.94 b	9.79 b	-2.13 a	0.44 e	1.96 c	7.52 b	3.01 b	46.24 d
Ebano	16.96 b	12.17 a	-1.90 a	0.76 a	1.16 g	4.87 e	2.83 d	55.78 a
Guarani	17.80 b	9.39 c	-2.65 b	0.33 g	1.74 e	7.00 c	2.95 c	44.58 e
Tupy	16.70 b	7.75 d	-3.24 c	0.27 h	1.66 f	7.52 b	2.96 c	35.78 f
Xavante	17.01 b	7.93 d	-3.23 c	0.44 e	1.80 d	7.52 b	2.93 c	52.37 b
Mean	17.72	9.34	-2.53	0.44	1.71	6.97	2.95	46.51
CV%	5.07	10.65	18.70	2.78	2.07	3.81	1.36	2.87

Means followed by the same letter in a column belong to the same group according to Scott-Knott at a 1% probability ($p \leq 0.01$).

Regarding the color, cultivars Arapaho, Cherokee and Choctaw presented the highest L values and were lighter fruits in relation to the fruits of the other cultivars that presented lower L values (Table 2). The Ebano and Choctaw cultivars were characterized by higher a★ values; visually, their fruits presented a more intense red coloration in relation to the other cultivars, thus the fruits displayed a more intense coloration. It is believed that the wide temperature range in a region of a tropical climate at an elevation highly influences the coloration of the fruits.

The vitamin C content varied from 42.69 mg 100 g^{-1} (Brazos) to 55.78 mg 100 g^{-1} (Ebano) (Table 2). Fruits with higher vitamin C contents are favored by the consumer, in spite of the fact that the quality attribute is not identified at the time of purchase.

Table 3 contains the Pearson correlation coefficients between the mineral nutrients and the quality indexes of the fruits of the different blackberry cultivars. The blackberry fruit quality variables presented little correlation with the macromineral contents. The best positive correlations were obtained between the minerals (P, Cu, Mn, Zn and Fe) with the soluble solid content and between the minerals (P, K, Mg, Cu and Zn) with the pH indexes. Regarding the negative correlations, higher values were observed between the color index b★ with the mineral manganese, the firmness with the minerals magnesium and copper and between the ascorbic acid content with the minerals copper and zinc.

There was a positive and significant correlation between the phosphorus content and the soluble solid characteristics, pH and titratable acidity (citric acid). The mineral phosphorus participates in some of the vital metabolic processes by supplying energy, increasing acid neutralization and sugar synthesis, resulting in less acidic fruits (KADER, 2008).

The potassium content correlated positively with the pH content and TTA and negatively with the firmness of the fruits. At adequate levels, potassium is frequently associated with the metabolism and concentration of carbohydrates, photosynthesis, enzyme activation, the soluble solid content, acidity and fruit firmness (MALAVOLTA et al., 1997; SUDHEER; INDIRA, 2007).

The contents of soluble solids, acidity, pH, minerals and color obtained in this work corroborates with the results obtained by Tosun et al. (2008) and the USDA (2005). However, the vitamin C content obtained in this work are low in relation to the values observed by Antunes et al. (2006) and superior to those found by Kwiatkowski et al. (2010) for the areas of Southern Minas and Southern Brazil, respectively. The variation in the nutritional quality of the fruits can be due to various biotic and abiotic factors but are possibly related to the light intensity and temperature range in the case of the present study (ALI et al., 2011; SIRIWOHARN et al., 2004; TAKEDA et al., 2002).

According to Hawkesford et al. (2002), calcium is the main element in the composition of the cell wall of the fruits, with the function of maintaining the integrity and permeability of the cell wall. However, a significant relationship between Ca and the firmness of the blackberry fruits was not observed, perhaps because the rupture of the cell wall and the liberation of Ca occur as a function of the ripening of the fruit.

A moderate negative correlation was observed between the calcium content in the blackberry fruits and the color index a★, which can be observed for the Ebano cultivar that presented redder fruits with lower calcium contents. The coloration is one of the most important attributes in the blackberry fruit choice by the consumer.

The minerals calcium, phosphorus and potassium combine with organic acids to influence the buffering capacity and, consequently, the perception of fruit acidity (KADER, 2008).

Magnesium is a major component of the chlorophyll molecule, and it participates in the photosynthesis process and aids in phosphorus absorption and translocation (HAWKESFORD et al. 2002). A positive correlation was obtained between the magnesium content present in the blackberry fruits and the L index, soluble solids and acidity. Due to its antagonistic action with phosphorus, a correlation exists in the firmness of the fruits.

Table 3. Pearson correlation coefficients between the quality attributes (L, a★, b★, firmness, vitamin C, total soluble solids - TSS, pH and total titratable acidity - TTA) and mineral contents of phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), copper (Cu), manganese (Mn), zinc (Zn) and iron (Fe) in the fruits of blackberry cultivars.

	P	K	Ca	Mg	Cu	Mn	Zn	Fe
L	0.11^{ns}	0.25^{ns}	0.21^{ns}	0.35^{*}	-0.01^{ns}	-0.26^{ns}	0.17^{ns}	0.19^{ns}
a★	-0.33^{*}	0.03^{ns}	-0.36^{*}	-0.05^{ns}	-0.26^{ns}	-0.44^{**}	0.08^{ns}	0.20^{ns}
b★	-0.11^{ns}	0.27^{ns}	-0.12^{ns}	0.24^{ns}	-0.18^{ns}	-0.51^{**}	0.02^{ns}	0.18^{ns}
Firmness	-0.63^{**}	-0.31^{*}	0.27^{ns}	-0.53^{**}	-0.67^{**}	-0.31^{*}	-0.42^{**}	0.06^{ns}
Vit. C	-0.52^{**}	-0.15^{ns}	0.34^{*}	-0.23^{ns}	-0.83^{**}	-0.30^{*}	-0.62^{**}	-0.23^{ns}
TSS	0.60^{**}	0.24^{ns}	0.30^{*}	0.37^{*}	0.62^{**}	0.51^{**}	0.61^{**}	0.55^{**}
pH	0.47^{**}	0.56^{**}	-0.04^{ns}	0.67^{**}	0.49^{**}	-0.26^{ns}	0.61^{**}	0.23^{ns}
ATT	0.46^{**}	0.48^{**}	0.28^{ns}	0.50^{**}	0.29^{ns}	0.26^{ns}	0.25^{ns}	0.52^{**}

ns, not- significant; *and **, significant at 5 and 1% probability, respectively, according to the Student's t test.

Copper is necessary for the synthesis of chlorophyll and functions as an activator of photosynthetic enzymes and respiration (MALAVOLTA et al., 2002). This element acts indirectly in the formation of organic acids through respiration and sugars through photosynthesis: the higher the respiration rate is, the higher is the cell wall degradation. Thus, copper was positively correlated with the attributes responsible for the flavor of the fruit and negatively with the texture.

Manganese has an important function in oxidation and reduction reactions and, being involved in carbon assimilation, is related primarily to photosystem I (MALAVOLTA et al., 2002). A negative correlation was observed among the Mn content with the colorimeter indexes a* and b* for the blackberry fruits. As the fruits in the green mature stage that present high chlorophyll contents reach the complete maturation stage, manganese is liberated as the chlorophyll is degraded, allowing the expression of anthocyanins.

The content of zinc correlates positively with the soluble solids and pH indexes and negatively with the vitamin C content and texture. According to Malavolta et al. (2002), the element zinc affects the synthesis and degradation of carbohydrates and participates in biochemical reactions that involve sugars.

As with the other micronutrients, the mineral iron is involved in chlorophyll-forming precursors and in the activation of enzymes involved in carbohydrate metabolism (MALAVOLTA et al., 2002). The effect of the micronutrient iron was pronounced, exhibiting significant correlation coefficients with the soluble solids and acidity index of the blackberry fruits cultivated in the Lavras, Minas Gerais State, region.

Conclusion

Differences were observed with regard to the quality of the fruits and the mineral content among blackberry cultivars produced in Southern Minas Gerais. 'Choctaw ' and 'Xavante ' presented the highest mineral contents. The fruits of the 'Caingangue' cultivar demonstrated the highest soluble solid content, and the Ebano cultivar demonstrated the highest vitamin C concentration and firmer, more intensely colored fruits, which were also observed in the fruits of the Choctaw cultivar. The quality of the fruits of the different blackberry cultivars presented little significant correlations with the macromineral content, whereas the opposite was obtained for the micronutrients.

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