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Blackberry Vinegar Produced By Successive Acetification Cycles: Production, Characterization And Bioactivity Parameters

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ABSTRACT

Blackberry vinegar was produced in successive acetification cycles and content of total phenolics, anthocyanins and antioxidant activity were evaluated along the production. Firstly, blackberry wine was obtained in bench-scale bioreactor, being verified 0.39 g/g ethanol yield, 1.78 g/L.h volumetric productivity and 76% efficiency. After, three successive acetification cycles were conducted efficiently in grapia barrel with average acetic acid production of 51.6 g/L, 72.2 % acetic acid yield and 0.4 g/L.h volumetric productivity. Appreciable contents of polyphenolic compounds, anthocyanins and high antioxidant activity were observed in the raw material, wine and vinegar obtained in each cycle of acetic acid transformation. Acetic acid transformation led the small reduction of antioxidant activity compared to alcoholic fermentation, but the antioxidant potential was maintained along the cycles. The content of total phenolics and anthocyanins also suffered a reduction in step of acetification.

Keywords: Berries, Acetic Acid Bacteria, Bioactives, Wine, Fermentation.

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INTRODUCTION

The production and consumption of berries has presented considerable growth in recent years, due to the increase in the interest of the consumers by nutritional benefits and functional, both of *in natura* fruit as the products processed the basis of berries (1).

Blackberry is one of the berries with more consumption in Brazil and in the world due to the peculiar taste, benefits to health and yet by the low calorific value (≅11 kcal/100 g) (2). The fruit is rich in vitamin C and mineral salts such as selenium, iron, calcium, magnesium, potassium and phosphorus, which are indispensable to the proper functioning of the organism. In addition, blackberry presents high content of polyphenols, ellagitannins, anthocyanins and antioxidant activity, when compared to other berries (3).

The consumption of blackberries has beneficial effects on the physical and mental health, especially due to the antioxidant potential of the anthocyanins and phenolic compounds present in large quantities in the fruit. Between the beneficial effects to health can be mentioned the protection against free radicals; reduction the risk of development of cardiovascular and coronary diseases; prevention of degenerative diseases; anti carcinogenic and anti-inflammatory effects (4,5).

The high post-harvest respiration rate and the fragility of the fruits reduces useful life, besides the fruit is quite susceptible to deterioration, mainly from fungal origin. In this way, products based on blackberry as frozen pulps, jellies, juices, ice cream, yogurt and teas are found with greater ease than the *in natura* fruit on the market (6,7).

The transformation of the fruit into wine and gourmet vinegar can be a good strategy for value aggregation and the strengthening of the productive chain. Wine and vinegar obtained from blackberries constitute products that allow and facilitate the consumption and use of bioactive compounds present in the fruit. Furthermore, the wide diversity of products containing vinegar such as sauces, ketchup, mayonnaise among others has stimulated the industrial production of this product (8).

In this context, the present work studied the processes of alcoholic fermentation and acetic oxidation conducted in (Brazilian gold wood) grapia barrel through successive cycles of acetic acid transformation. The wine and vinegar produced were characterized by physical-chemical parameters of quality, total phenolics content, anthocyanins and antioxidant activity. In addition, the total phenolics content, anthocyanins and the antioxidant potential of *in natura* fruit, wine and vinegar were compared among themselves.

MATERIALS AND METHODS

Raw material and process of bioactive compounds extraction

Blackberry (*Rubus sp*. var. guarani) was acquired from the farm producing fruits located at Southwest region of Paraná, Brazil. The fruits were kept frozen (-18 °C) until processing.

Anhydrous ethanol and acetone were evaluated as extracting agents of phenolic compounds and antioxidants of fruit. Blackberries *in natura* were triturated and submitted to extraction with the extracting agent in Erlenmeyer flasks using the proportion of 5 g of fruit for 20 mL of solvent. The flasks were kept at 25 ºC under stirring of 150 rpm for 5 minutes in rotatory shaker. The extracts were separated from the pulp and skins by filtering on filter paper and employed in analyzes of total phenolics, anthocyanins and antioxidant activity.

Microorganisms and inoculums cultivation

Saccharomyces cerevisiae f.r. *bayanus* (Fermol, Perlage, AEB Biochemistry Latin American SA) was cultivated in malt extract medium (20 g/L malt extract, 1 g/L peptone and 20 g/L glucose) for 24 h (120 rpm, 28 ºC). The yeast cells were recovered from the medium by centrifugation (1350 x g, 30 min.) and re-suspended in saline solution (0.9% w/v). Standardized pre-inoculum containing 2 x 10^6 cells/mL was employed in the alcoholic fermentation process.

Mixed culture of acid acetic bacteria was isolated from colonial red grapes vinegar (non-pasteurized). GY medium (10% glucose, 1% yeast extract and 100 mg/L natamycin) was employed in the process of acetic culture selection and insolation. A volume of 10 mL of non-pasteurized vinegar was transferred for Erlenmeyer flasks containing 100 mL of GY medium and incubated for 48 h at 30 ºC and 120 rpm. Acetic culture was recovered by centrifugation (1350 x g, 30 min) and resuspended in physiological saline solution (0.9% w/v) until obtaining a solution with 0.5 nm optical density at

600 nm. Inoculum was prepared by the mixture of 25 mL culture acetic and 155 mL blackberry wine in 250 mL Erlenmeyer flasks. The flasks were kept in orbital shaker for 24 h at 30 ºC and 120 rpm for growth and cell adaptation.

Alcoholic fermentation and acetic oxidation

Blackberry fruits were slowly thawed under refrigeration, and then depulped in multiprocessor and passed through (mesh 0.5 mm) stainless steel sieve for removal of seeds and part of husks. Content of soluble solids was corrected to 18 °Brix with commercial sucrose. The must was then supplemented (30 g/hl) by the addition of yeast growth activator (ENOVIT®, Pascal Biotech, France) and sulphited by addition of 50 mg/L of sodium metabisulphite.

Pre-inoculum of *S. cerevisae* (35 mL, 2 x 10^6 cells/mL) was transferred to 5L fermentation vessel containing 315 mL of most and cultivated in bench-scale bioreactor (Biostat B, B. Braun Melsungen, Germany) for 24 h at 28 ºC. The alcoholic fermentation was initiated by the addition of 3150 mL of must to bioreactor containing adapted inoculum and conducted at 28 ºC.

Blackberry wine (1620 mL) obtained from alcoholic fermentation was supplemented with Acetozym® (Heinrich Frings GmbH & Co, USA), transferred to grapia barrel (Brazilian gold wood) and inoculated with 180 mL of inoculum. The inoculated wine was maintained at 30 °C in cell culture incubator. Were conducted three successive cycles of acetic acid transformation with withdrawal of 65% of the vinegar produced and addition of an equal volume of wine to each cycle (acidity between 4.0-4.5%). Vinegars produced were centrifuged (1350 x g, for 30 minutes), filtered on filter paper, bottled and pasteurized for later characterization.

Physical-chemical characterization and bioactivity parameters

Blackberry was characterized as regards the content of lipids (Soxhlet extraction method), crude protein (Kjeldahl method), mineral residue (incineration at 550 ºC), crude fiber, moisture (kiln-drying method at 105 °C), total soluble solids (hand refratometer), pH, titratable acidity (titrimetric method) according the Association of Official Methods of Analytical Chemists (9). Total reducing sugars was determined by DNS method after hydrolisys with HCl 1 mol/L (10), total phenolic compounds by spectrophotometric Folin-Ciocalteau method (11), total anthocyanins content by pH differential method (12) and antioxidant activity using DPPH method (13) and $ABTS^+$ cation radical discoloration assay (14).

The wine and vinegar of blackberry were submitted to analysis of pH, titratable acidity, total soluble solids, density, total and free sulfur dioxide content, total dry extract and reduced, sulphate (9), total reducing sugar (DNS method) and total phenolic compounds, anthocyanins and antioxidant activity as described above. Ethanol and acetic acid were measured by HPLC using refractive index (IR) detector and Bio-Rad HPX-87-H (300 7.8 mm) column at 45ºC, 0.005 mol/L sulphuric acid the eluent, flow rate of 0.4 mL.min) and sample volume of 20 µL.

RESULTS AND DISCUSSIONS

Physical-chemical parameters of blackberry fruits

The results of the physical-chemical analysis and bioactivity parameters of blackberry samples are shown in Table 1. The pH value of fruits was 3.22 and the titratable acidity expressed in citric acid was 1.51 g/100 g. Similar results were described by Souza et al. (15), which found similar physical-chemical characteristics between berries grown in tropical regions of Brazil and berries cultivated in zones with temperate areas.

On the other hand, the moisture content found (58.5 g/100g) in the sample analyzed was lower than that described by these authors, which described values of 87.92 g/100g. The lower moisture content found is possibly associated with the loss of water by syneresis during thawing of fruits. Low protein content (1.47 g/100g) and lipids (0.21 g/100g) were also checked and are in concordance with the literature data (15).

Relatively high content of mineral residue (2.17 g/100g) and fibers (14.33 g/100g) were found and such results are associated with the presence of bark and some seeds in the analyzed samples. The content of total reducing sugars was 9.8 g/100g and solids soluble content was 9 ºBrix, which are within the average values reported by other authors (16,17).

In the literature are reported the use of different solvents for the extraction of polyphenols from fruits, as acidified methanol (18), ethanol (19), acidified ethanol (20); acetone (19) and acidified acetone (17). In this study, ethanol and acetone

were evaluated as extracting agent of bioactive compounds.

As can be seen in Table 1, in a general way, acetone provided better extraction of phenolic compounds, with an efficiency 73.1% higher (1.702 mg GAE/100g) than obtained with the ethanol (983.4 mg GAE/100g). This behavior was also described by Vizzotto and Pereira (21) and Ubeda et al. (22), which obtained better results of extraction of phenolic compounds of blackberry and strawberries using acetone as extracting agent.

Souza et al. (15) used solution of methanol and water (50:50 v/v) for the extraction of phenolic compounds from blackberries and found a maximum content of 850.52 mg GAE/100g, lower result than that obtained in this work both in extraction with ethanol (983.4 mg GAE/100g) as with acetone (1.702 mg GAE/100g).

With relation to the levels of total anthocyanins present in fruit, were also verified larger contents when employed the solvent acetone (511.7 mg cyanidin-3-glucoside/100g) in the protocol of extraction. This content was 28.13% higher than that obtained with extraction in ethanol (399.4 mg cyanidin-3-glucoside/100g). In accordance with Siriwoharn and Wrolstad (23) the content of anthocyanins, in particular the cyanidin-3-glucoside that is the majority of the blackberry pigment, varies considerably depending on the stage of maturation of the fruit. These authors reported lower content to the obtained in this study (317 mg cyanidin-3-glucoside/100g).

Similarly to that observed with the total phenolic content and anthocyanins, the solvent employed in the extraction process, influenced the values of DPPH and $ABTS^+$ radical scavenging capacity. Values of 38.5 µmol Trolox equivalent /g (TE/g) were observed when performed extraction with acetone and 36.01 µmol TE/g when employed ethanol. Such values statistically differed among themselves ($p < 0.05$), indicating that the acetone promoted better extraction of substances with antioxidant activity measured by the DPPH method.

In relation to the antioxidant activity assessed by the $ABTS^+$ radical scavenging method, no significant difference was observed in the values obtained with both solvent extractors (9.43 µmol TE/g fruit in acetone and 9.16 µmol TE/g fruit in ethanol). This result suggests that ethanol and acetone showed similar capacity of extraction of antioxidant compounds with similar antioxidant potential measured through this method ($ABTS^+$ radical scavenging).

Table 1. Physical-chemical parameters, total phenolics, anthocyanins and antioxidant activity of blackberry fruits.

Physical-chemical parameters	**Observed values**	
pH	3.22 ± 0.002	
Titratable acidity (g/100g)	1.51 ± 0.002	
Moisture (g/100g)	58.05 ± 0.70	
Crude protein (g/100g)	1.47 ± 0.49	
Lipid (g/100g)	0.21 ± 0.03	
Mineral residue (g/100g)	2.17 ± 0.11	
Crude fiber (g/100g)	14.33 ± 1.53	
Reducing sugars (g/100g)	9.81 ± 0.00	
Total soluble solids (°Brix)	9.00 ± 0.00	
	Extracting solvent	
	Acetone	**Ethanol**
Total phenolics (mg GAE/100g)	1702.0 ± 7.27[a]	983.4 ± 5.21[b]
Anthocyanins (mg cyanidin-3-glucoside/100g)	511.7 ± 0.0[a]	399.4 ± 0.0[b]
Antioxidante Activity-DPPH (µmol TROLOX equivalent/g)	38.5 ± 0.47[a]	36.01 ± 0.51[b]
Antioxidante Activity- $ABTS^+$ (µmol TROLOX equivalent/g)	9.43 ± 0.14[a]	9.16 ± 0.11[a]

[a,b]Different letters, in the same line, are significantly different to each other ($p < 0.05$).

GAE: gallic acid equivalent.

Alcoholic fermentation

The fermentative profile of must based on blackberry pulp is shown in the graph in Figure 1. The yeast used in process has shown high fermentative capacity with ethanol accumulation of 63.9 g/L (corresponding to 8.09%, v/v) after 36 hours of fermentation and 76% substrate consumption. Similary, Hong-Guang et al (24) reported concentrations of ethanol ranging from 3 to 7.4% in alcoholic fermented beverage produced from must based on blackberry chaptalized with sucrose (15 ºBrix).

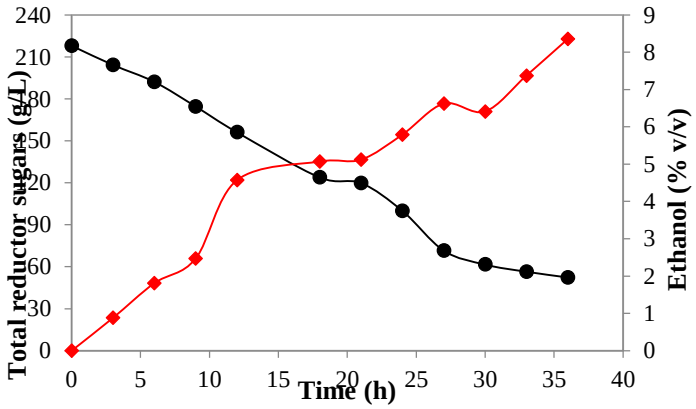

Figure 1 - Content of total reducing sugars (•) and ethanol (♦) along the alcoholic fermentation.

Lag phase was not observed in the alcoholic fermentation (Fig. 2). It was observed linear cell growth up to 10 hours of cultivation, which was accompanied by 20% substrate consumption. The behavior of the yeast related to the consumption of substrate and cellular growth indicates that the same presented good metabolic activity along of fermentation and that the inoculum was adequately prepared and adapted.

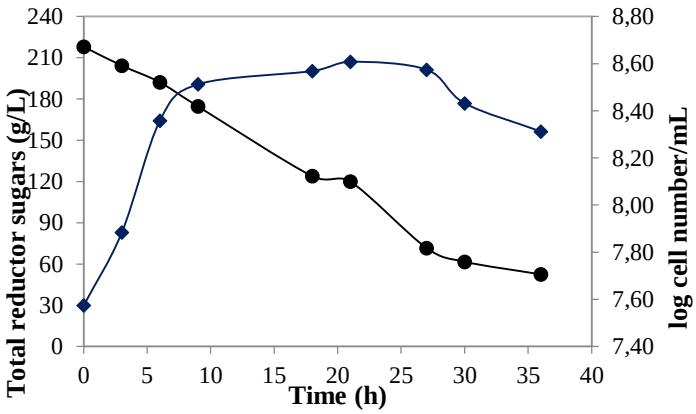

Figure - 2 Content of total reducing sugars (•) and cell concentration (♦) along the alcoholic fermentation.

After 36 hours of cultivation there was stabilization of the soluble solids content in fermentation must and stopped the detachment of carbonic gas bubbles, indicating the end of fermentation. In the literature are checked studies that describe the different times of alcoholic fermentation of fruit juices (25–27). Coelho et al. (28) using different fruit for wine production, described the times that varied from 40 h (cherry) to 190 h (orange).

At the end of the alcoholic fermentation was verified ethanol yield ($Y_{P/S}$, ratio between final ethanol concentration and sugar consumption

during the run) of 0.39 g/g, volumetric productivity in ethanol (Q_P, ratio between final ethanol concentration and fermentation time) of 1.78 g/Lh, average rate of substrate consumption (Q_S, ratio of the glucose consumed to the fermentation time) of 4.6 g/Lh, efficiency fermentative (η, ratio of the observed ethanol yield to the theoretical yield of ethanol (0.511 $g.g^{-1}$) of 76% and a substrate consumption percentage of 76%. The incomplete consumption of sugars present in must of fermentation can be associated to the accumulation of ethanol and consequent inhibition of metabolic activity (inhibition by formed product). Another aspect that may have contributed to the permanence of a sugar residual in must is the possible exhaustion of nutrients (mineral and source of nitrogen) along the fermentation, once that were not added nutrients during the fermentation process, but only at the beginning of the process.

Characterization of blackberry wine

Blackberry wine presented pH 3.18, titratable acidity expressed in acetic acid of 0.92 g/100 mL and total soluble solids content of 6 °Brix, as described in Table 2. Similar results were described by Oliveira et al. (29) when preparing alcoholic fermented beverage of cagaita (*Eugenia dysenterica* DC), which found pH value of 3.28 and contents of soluble solids of 5.77 °Brix.

The density of blackberry wine was 1052.8 g/L. This parameter varies according to the quantity of sugar and ethanol present in fermented. In fact, the blackberry wine presented relatively high content of residual sugars (52.4 g/L), which reflected in the density value.

With relation to the content of free and total sulfur dioxide, values observed were of 15.32 mg/L and 40.96 mg/L, respectively. Such results are in accordance with the limits recommended by the Brazilian legislation, establishing a maximum value of 200 mg/L of total SO_2 in wines of fresh fruit (30).

High total phenolics content was verified in the wine (199.25 mg GAE/L), however, it is noticeable reduction of such content when compared to values found in fruit *in natura* (1702.0 and 983.4 mg GA/100g, Table 1). This reduction may be related to the fact of the blackberry must have been formulated with the pulp of the fruit, without seeds and with reduced quantity of husks. In fact, the largest contents of polyphenols are commonly found in the husks and seeds on the fruit.

Budak and Guzel-Seydim (14) when evaluated the content of total phenolic compounds in wine derived from Ulugbey Karasi grapes produced in Turkey, found values significantly below (237 mg GAE/L). On the other hand, Su and Chien (31) verified superior results to those obtained in this study, describing the content of 858 mg GAE/L in blueberry wines fermented without bark and 1150 mg GAE/L in fermented wines with bark. Such results indicate that the presence of bark in must during alcoholic fermentation, can contribute to greater content of total phenolic compounds in wine produced.

Similarly to that observed with the content of polyphenols, occurred decrease in the content of anthocyanins present in the wine (51.93 mg cyanidin 3-glucoside /L) in relation to fruit (511.7 and 399.4 mg cyanidin 3-glucoside /L), as shown in Tables 1 and 2. In the same way, Mena et al. (32) reported content between 1360 mg cyanidin 3-glucoside /L and 230 mg cyanidin 3-glucoside /L in pomegranate juice from variety wonderful and Mollar Elche, however, when carrying out the winemaking such compounds have reduced 46% and 61%, respectively.

The tests for assessing free radicals scavenging capacity by methods DPPH and $ABTS^+$ demonstrated that the blackberry wine presents antioxidant activity. The antioxidant activity of the product is associated with the presence of polyphenolic compounds, flavonoid and anthocyanins, among other bioactive compounds from the fruit.

Blackberry wine presented an antioxidant potential, measured in trolox equivalent, of 139.52 µmol TE/L by the DPPH method and 21.24 mmol TE/L by the $ABTS^+$ method. Budak and Guzel-Seydim (14) verified lower values in red grapes wine from variety Ulugbey Karasi, when assessing the antioxidant capacity by the $ABTS^+$ method. Such authors describe values of 11.20 mmol TE/L. On the other hand, Mulero et al. (33) to evaluate the antioxidant activity (ABTS method) of wines from traditional and organic grapes observed activities of 6.78 mmoL TE/mL in organic grapes wine and 6.02 mmol/mL in traditional grapes wine.

Acetic oxidation and characterization of blackberry vinegar

As can be seen in Figure 3, the inoculum composed of acetic acid bacteria isolated from colonial vinegar, demonstrated efficiency in conversion of ethanol to acetic acid. Good microbial performance was verified over the three successive cycles of alcohol-acetic acid transformation.

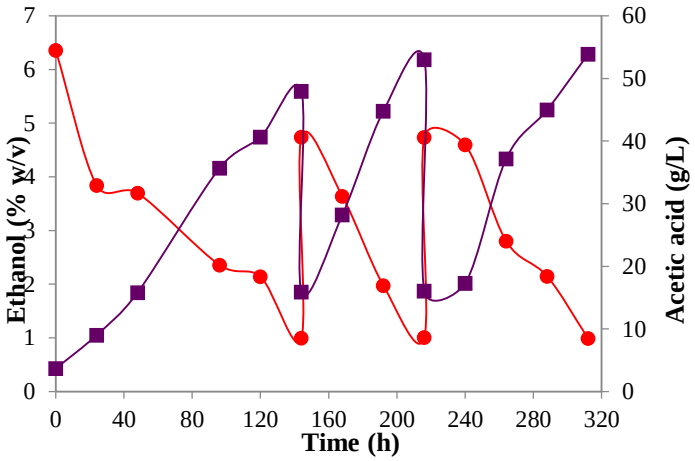

Figure 3 - Content of ethanol (•) and acetic acid (■) along the cycles of acetic acid transformation.

The first cycle had duration of 144 h, when ethanol consumption was of 85% of the content present in wine and the acetic acid production was of 47.9 g/L (Fig. 3 and Table 3). Already in the second cycle, considerable reduction in time of conversion was observed (72 h) and production of 52.9 g/L acetic acid and 78.8% substrate consumption.

The reduction in the fermentation time in 50% in the second cycle in relation to the first may be associated to a higher amount of acetic acid bacteria inside the barrel. In fact, in the second cycle of fermentation, had already complete formation of superficial film, known as mother of vinegar. This film is formed by polysaccharides and facilitates the contact of acetic acid bacteria with superficial oxygen. Possibly in the first cycle of fermentation, due to absence of this initial film, which is formed along the process of acetic acid transformation, there was lower efficiency in acetic oxidation by bacteria.

However, in the third cycle time of acetic acid transformation increased to 96 hours with production of 53.84 g/L acetic acid and 79.1% substrate consumption. Possibly, the increase in the time of conversion in the third cycle in relation to the second, may be associated with possible excessive accumulation of biomass and acetic acid bacteria in superficial gelatinous layer with a consequent reduction in the transfer and absorption of oxygen by acetic acid bacteria, having as a consequence some reduction in oxidative conversion of ethanol to acetic acid. It is important to highlight that in the vinegar production the availability of oxygen is a limiting factor in the process of acetic acid transformation (34).

A physical-chemical parameter of great importance to the quality of vinegars is the acidity. Vinegars with acidity less than 4% may suffer anguillulas infections (vinegar eels) and on the other hand, vinegar with acidity exceeding 5.5 % can submit as products very acid and are rejected by the consumers. In the present study, the acidity varied from 4.09 g/100 mL in the first cycle to 4.91 g/100 mL in the third cycle of acetic acid transformation, values that indicate adequate acetic acid transformation and obtaining product with good acidity.

The total soluble solids content remained constant in 3 cycles of acetic acid transformation (5 ºBrix), being verified small reduction of solids in relation to blackberry wine (6 ºBrix). Such reduction in the levels of soluble solids may be related to the possible consumption of residual sugars present in alcoholic fermented (wine) during the acetic oxidation.

Brazilian legislation establishes that fruit vinegars must contain at least 1 g/L and a maximum of 5 g/L of fixed mineral residue; in this sense all vinegar produced present suitable values. The total dry extract parameter demonstrates the content of mineral and organic materials that remain after the evaporation of water and other volatile substances of vinegar. The values found are consistent with vinegar produced the basis of fruit pulps and evidence the quality of vinegar obtained.

With relation to the content of sulphates, vinegars of fruit must contain at most 1 g/L (35). The values found in the three consecutive batches were well below (0.026 g/L), being therefore within the limit established by legislation. Another important aspect

and defined by the Brazilian legislation is that fruit vinegars must not exceed 1% (v/v) alcohol content. According to data described on Table 3, the values obtained in three cycles are in line with the legislation (between 0.93 and 0.95 g/L).

Considerable quantities of phenolic compounds were verified in the vinegar obtained in three cycles of acetic acid transformation (138.95 mg GAE/L; 151.8 mg GAE/L and 165.2 mg GAE/L). It is interesting to note that there was little reduction of total phenolics content derived from of wine (199.25 mg GAE/L, Table 2) in vinegars produced. The content of phenolic compounds present in vinegars indicates that there was no significant losses of such compounds by oxidation during acetic acid transformation. In this sense, Ubeda et al. (22), point out that the limited exchange of oxygen during acetic acid transformation by slow process of acetic acid transformation (Orleans method), contributes to minors losses of such compounds by oxidation in relation to processes that use injection of air.

On the other hand, a noticeable reduction of phenolic compounds in wine compared to the quantities originally present in fruit was verified (ethanolic extract: 983.4 mg GAE/100g; acetone extract: 1702.00 mg GAE/100g). The reduction of the content of the phenolic compounds in wine in comparison to that seen in fruit, can be justified by the fact of fruit have been analyzed in its full form, i.e. with bark and seeds that are parts of fruit rich in phenolic substances.

Similarly, there was a reduction of total anthocyanins content on wine (51.93 mg cyanidin-3-glucoside/L) in relation to the one verified in fruit (511.7 and 399.4 mg cyanidin-3-glucoside/100g). It was also verified reductions of anthocyanins content (1st cycle: 49.8%; 2nd cycle: 34.5% and 3rd cycle: 14%) in vinegar obtained in different cycles with regard to wine.

However, although it was observed a reduction of total anthocyanins content, the contents present in vinegar are appreciable (1st cycle: 26.05; 2nd cycle: 34.0 and 3rd cycle: 32.78 mg cyanidin 3-glucoside /L). Su and Chien (31) verified lower anthocyanins content in blueberry vinegar produced from fermented wines without bark (9.7 mg cyanidin 3-glucoside /L) and with bark (32.2 mg cyanidin 3-glucoside/L).

Several studies evaluating the antioxidant capacity of different fruits are reported in the scientific literature. However, there are few reports evaluating the antioxidant potential in fruit vinegars. The results of the tests of antioxidant activity *in vitro*, described in Table 3 demonstrate that the blackberry vinegars showed significant ability to scavenge DPPH and $ABTS^+$ radicals. Similar values of DPPH radical scavenging ability were verified in the vinegar produced in three cycles of acetic acid transformation (1st cycle: 103.5 µmol TE/mL, 2nd cycle: 107.35 µmol TE/mL and 3rd cycle: 107.73 µmol TE/mL). Such values are higher than those found by Budak and Guzel-Seydim (14) in grape vinegar (13.50 µmol TE/mL). Ubeda et al. (22) described values between 3227 µmol TE/kg and 3388 µmol TE/kg in strawberry vinegar.

The $ABTS^+$ radical scavenging ability verified in three samples of vinegars were also similar between themselves. In the first cycle, the antioxidant activity front to the radical $ABTS^+$ was 15.63 mmol/L, in the second was 17.36 mmol/L and in the third cycle was 19.03 mmol/L.

Comparing the results of the DPPH and $ABTS^+$ radicals scavenging ability of wine and vinegar produced is verified some reduction in the antioxidant potential. There was a reduction in DPPH radical scavenging ability between 22.8% (3rd cycle) and 25.8% (1st cycle). With relation to $ABTS^+$ radical scavenging ability the percentage in the reduction of antioxidant activity varied from 10.4% (3rd cycle) and 26.4% (1st cycle).

CONCLUSIONS

The blackberry fruits studied presented commercial quality and physical-chemical characteristics similar to cultivars produced in other Brazilian regions. The fruits are highlighted by nutritional values, especially by the content of sugars, fibers, total phenolics and antioxidant potential. The high content of sugars make the blackberry an attractive fruit for the winemaking and production of vinegar and obtaining such products can circumvent problems of loss due to high perishability and fragility of the fruit.

Industrial yeast used in alcoholic fermentation has shown efficiency in the process, being produced blackberry wine with pleasant aroma and taste. Were verified alcoholic fermentation yield of 0.39 g/g, volumetric productivity in ethanol of 1.78 g/L.h and efficiency of 76%. Inoculum composed of acetic acid bacteria isolated from colonial vinegar was effective in acetic acid transformation, with maintenance of the efficiency of the process in successive cycles of acetic acid transformation.

Vinegars produced presented physical-chemical parameters of quality consistent with Brazilian legislation, as well as appreciable contents of phenolic compounds, anthocyanins and antioxidant potential.

The production of blackberry vinegar in wood barrel (slow process) can be a good option of income for family producers, considering the hardiness of fruit cultivation, the simplicity of the vinegar production process and the possibility of value aggregation to the productive chain.

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A N I N T E R N A T I O N A L J O U R N A L

Erratum

In Article "Blackberry Vinegar Produced By Successive Acetification Cycles: Production, Characterization And Bioactivity Parameters", with the number of DOI: http://dx.doi.org/10.1590/1678-4324-2016150136, published in journal Brazilian Archives of Biology and Technology, vol. 59, the 06 page.

To include:

Table 2. Physical-chemical characterization and bioactivity parameters of blackberry wine.

Physical-chemical parameters	Observed values
pH	3.18
Titratable acidity (g/100 mL)	0.92± 0.004
Total SolubleSolids (°Brix)	6 ± 0.00
Ethanol (%, v/v)	8.9± 0.1
Total reducing sugar (g/L)	52.4 ± 0.002
Density at 20 ºC (g/mL)	1052.8 ± 0.00
Free sulfur dioxide - SO_2 (mg/L)	15.32± 0.002
Total sulfur dioxide - SO_2(mg/L)	40.96± 0.004
Total phenolic compounds (mg GAE/L)	199.25 ± 2.19
Anthocyanins (mg cyanidin-3-glucoside/L)	51.93 ± 0.53
Antioxidant activity - DPPH (µmol TE/mL)	139.52 ± 7.07
Antioxidant activity - ABTS (mmol TE/L)	21.24 ± 1.24

GAE: gallic acid equivalent

TE: trolox equivalent

In the 08 page, to include:

Tabela 3. Physical-chemical characterization and bioactivity parameters of blackberry vinegar produced in barrel of brazilian gold wood.

Parameters analyzed	Observed values		
	1st cycle	2nd cycle	3rd cycle
Acetic acid production (g/L)	47.9 ± 5.0[a]	52.9± 1.82[a]	53.84± 0,31[a]
Ethanol consumption (%)	85± 1.86[a]	78.8± 5.28[a]	79.8± 2.42[a]
pH	2.62 ± 0.0[b]	2.63 ± 0.0 [b]	2.7 ± 0.01[a]
Titratable acidity (g/100mL)	4.09 ± 0.08[c]	4.53 ±0.02[b]	4.91 ± 0.41[a]
Total soluble solids (°Brix)	5.0 ± 0.0[a]	5.0 ± 0.0[a]	5.0 ± 0.0[a]
Mineral residue (g/L)	3.73 ± 0.0[b]	3.9± 0.05[a]	3.74 ± 0.0[b]

Total dry extract (g/L)	44.3± 0.04[a]	33.3 ± 0.05[b]	32.0 ± 0.21[c]
Dry reduced extract (g/L)	15.68± 0.06[c]	26.69 ± 0.06[b]	26.98 ± 0.01[a]
Density at 20 ºC (g/mL)	1077.3± 0.11[a]	1077.8 ± 0.05[a]	1077.7 ± 0.05[a]
Sulphates (g/L)	0.026 ± 0.0[a]	0.026 ± 0.0[a]	0.026 ± 0.0[a]
Ethanol (g/L)	0.95 ± 0.08[a]	0.94 ± 0.090[a]	0.93 ± 0.07[a]
Total phenolics (mg GAE/L)	138.95[b]	151.8[b]	165.2[a]
Anthocyanins (mg/L)	26.05± 0.35[a]	34.23 ± 0.46[a]	32.78 ± 0.56[a]
Antioxidant activity - DPPH (µmol TE/mL)	103.5± 2.35[a]	107.35± 5.95[a]	107.73± 5.95[a]
Antioxidant activity - ABTS (mmol TE/L)	15.63 ± 0.95[c]	17.36 ± 0.99[b]	19.03 ± 0.99 [a]

[a,b,c]Different letters, in the same line, are significantly different to each other ($p < 0.05$).
GAE: gallic acid equivalent, TE: trolox equivalent.