



CHEMICAL SCIENCES

Solvent-free sonication of blackberries for the anthocyanin enrichment of juices obtained by pressing

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Abstract: An ultrasound pretreatment was used to increase anthocyanins content in blackberry juice. Whole fruits were inserted into a glass vessel without contact with any solvent, sonicated in an ultrasonic bath, and then pressed with a manual juicer. The experimental design showed that 7 min at 65% of ultrasound amplitude increased the anthocyanin content in juices from 31 to 56% for BRS Xingu, Guarani, and Xavante cultivars. Two major anthocyanins, cyanidin-3-glucoside and cyanidin-3-rutinoside were found in higher concentrations for sonicated fruits. Therefore, ultrasonic pretreatment of whole fruits increased the anthocyanins in blackberry juices using a simple, fast, and green approach.

Key words: bioactive compounds, blackberries, fruit juice, phenolic compounds, ultrasound.

INTRODUCTION

Blackberry is a small berry fruit known to have compounds with relevant bioactive activity, such as phenolic ones (Schulz & Chim 2019). Anthocyanins are the class of phenolic compounds most abundant in blackberries (Moraes et al. 2020), responsible for the beneficial properties related to the ingestion of these fruits (Gowd et al. 2019). Some studies have shown that anthocyanins from blackberries presented positive effects on cancer (Bowen-Forbes et al. 2010, Dai et al. 2009), inflammation (Bowen-Forbes et al. 2010), diabetes (Gowd et al. 2019), and neuroprotection (Chaves et al. 2020). Therefore, the consumption of such compounds from blackberries could be related to benefits for health (Bowen-Forbes et al. 2010).

Despite these benefits, blackberries are perishable due to the high moisture content and

low resistance to physical damage, and juicing is commonly used for processing (Tiwari et al. 2009). Conventionally, fruit juices are obtained through mechanical pressing, which maintains the functional and sensory properties of the fruit by processing at room temperature (Renard & Maingonnat 2012). However, pressing is not exhaustive because it provides only a partial transfer of intracellular components from fruits to the juice (Wang et al. 2019).

Thus, new approaches have been used to increase the content of bioactive compounds in fruit juices (Wang et al. 2020). Pérez-Grijalva et al. (2018) used microwaves to blanch blackberries before juicing by centrifugation, increasing the content of anthocyanins in the juice around three times in comparison to the control. However, microwave heating should be carefully applied to avoid high temperatures

and the degradation of phenolic compounds. Some phenolic compounds can resist 60-80 °C, but anthocyanins are more vulnerable to high temperatures, degrading at temperatures above 45 °C (Antony & Farid 2022).

Ultrasound has been used at room or mild temperatures to improve the extraction of bioactive compounds (Chemat et al. 2017). The cavitation generated by sonication promotes ruptures of the membrane and cell wall, resulting in the leakage of the intracellular content of plant cells (Bora et al. 2017). Studies using an ultrasound probe for the treatment of fruit juices showed that the content of phenolic compounds, including anthocyanins, was increased when compared to juices not treated. (Guerrouj et al. 2016, Ordóñez-Santos et al. 2017, Wang et al. 2019, 2020). It was found to increase anthocyanins content by 20% in barberry juice (Radziejewska-Kubzdela et al. 2020), 25% in sohiong juice (Vivek et al. 2019), 4.2% in bayberry juice (Cao et al. 2019) and 24% in cranberry juice (Gomes et al. 2017). However, only the ultrasonic treatment after juicing was performed in these works, and no results were found for the sonication of whole fruit as a pretreatment before juicing.

Therefore, there is a lack of studies on the efficiency of ultrasound before juicing. In this way, the whole fruit, without contact with any solvent, was sonicated before pressing to increase the anthocyanins content of the blackberry juice. The fruits were inserted into a glass vessel for sonication in an ultrasonic bath. The experimental conditions (time and ultrasound amplitude) were optimized by central composite rotational design (CCRD). The anthocyanin profiles of juices obtained by pressing with and without ultrasound were compared.

MATERIALS AND METHODS

Samples

For experimental design, blackberry fruits (*Rubus* sp., unidentified cultivar) were collected in Jaguari city, RS, Brazil. After optimizing experimental parameters for the ultrasound pretreatment, blackberries from the BRS Xingu, Guarani, and Xavante cultivars were obtained from Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA) Clima Temperado located in Pelotas city, RS, Brazil and used in the experiments. All fruits were stored (-18 °C) until the juice preparation. Portions of about 60 g of fruit were thawed to room temperature inside a 500 mL glass Erlenmeyer before the experiments.

Pretreatment with ultrasound

The Erlenmeyer containing 60 g of the thawed whole blackberry fruits was placed in the center of the ultrasound bath (model D-78224, 25 kHz, 100 W, Elmasonic, Elma, Germany). The maximum level allowed (3.5 liters) for the ultrasonic bath was used, and the temperature was maintained at 25 ± 2 °C with the addition of cold water if necessary. The ultrasound amplitude and sonication time were optimized as described in the *Experimental design and statistical analysis* section. The procedures used to obtain blackberry juice are outlined in Figure 1.

Juice extraction

The juices were obtained by pressing 60 g of fruits with a manual aluminum juicer (22 x 9 x 9 cm, Alumina, Belo Horizonte, Brazil). The fruits were pressed until no more juice was removed. The juices were placed in 50 mL Falcon tubes and centrifuged at 3000 rpm for 5 min to remove particles, and the supernatant was then immediately analyzed.

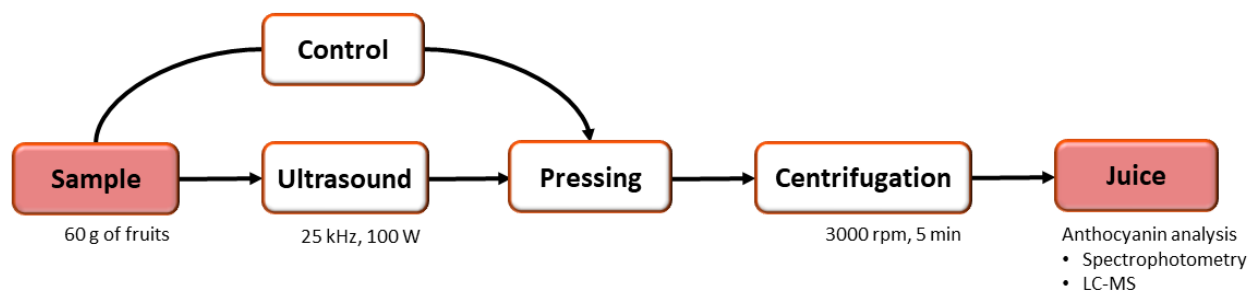


Figure 1. Procedures used to obtain blackberry juice.

Determination of total monomeric anthocyanins

The concentration of total monomeric anthocyanins was determined using differential pH methodology (Giusti & Wrolstad 2001) in a spectrophotometer (model 8453, Agilent, Santa Clara, United States) at 520 and 700 nm wavelengths. The values were reported in mg of cyanidin-3-glucoside eq./L of juice.

Analysis of individual anthocyanins by liquid chromatography mass spectrometry (LC-MS)

Anthocyanins were identified in the blackberry juices using a liquid chromatography mass spectrometer (LCMS 8045 equipment, Shimadzu, Kyoto, Japan) with electrospray source ionization (ESI). The samples were previously purified in solid-phase extraction (SPE) cartridges containing octadecyl silica (Stracta C18-E, 500 mg/6 mL, 55 μm particle size) before injection into LC-MS (Giusti & Wrolstad 2001). A C18 column (Zorbax Eclipse XDB, 150 mm, 2.1 mm, 3.5 μm particle size) was used for the separation of the compounds, using the methodology described by Barcia et al. (2014) with modification by Farias et al. (2022). Two solvents were used for the separation: the mobile phase A consisting of water, acetonitrile, methanol, and formic acid (88.5:3:8:0.5, v/v/v) and the mobile phase B consisting of water, acetonitrile, methanol, and formic acid (41.5:50:8:0.5, v/v/v). The flow was 0.19 mL/min, with an elution gradient of 46 min which was defined as follows: 3% B (0-8 min),

30% B (8-28 min), 50% B (28-34 min), 100% B (34-40 min), 3% B (40-46 min). For the separation, 10 μL of samples were injected into the LC with an oven temperature of 40 $^{\circ}\text{C}$.

The positive mode was used through the electrospray ionization source (ESI), with mist gas flow 2.0 L/min, drying gas flow 4.0 L/min, heating gas flow 6.0 L/min, voltage interface 4.9 kV, interface temperature 346 $^{\circ}\text{C}$, temperature DL 163 $^{\circ}\text{C}$, heat block temperature 200 $^{\circ}\text{C}$, dynode voltage 10 kV and detector voltage 1.78 kV. The optimization of multiple reaction monitoring (MRM) for the cyanidin-3-glucoside and perlagonidin-3-glucoside standard was performed as described by Farias et al. (2022). The other anthocyanins were identified from the values of mass/charge (m/z) because there was no standard available. Thus, a process of identification attempt was performed considering the data obtained from the literature in which the anthocyanins from blackberries were analyzed (Moraes et al. 2020). Thus, the compounds identified in this work were added to the methodology of quantification and identification of anthocyanins by LC-MS/MS through their mass/charge (m/z) values. In addition, from the knowledge of chromatograms of previous works of our group and the literature, as well as the order of elution of the compounds in the separation process on the chromatographic column, it was also possible to perform the tracking of the peaks (Moraes et al. 2020). More details about the optimization of

the method are described by Farias et al. (2022). The quantification was carried out through a cyanidin-3-glucoside calibration curve with seven points and with concentrations from 0.25 to 3.25 mg/L and $R^2 = 0.997$. The results were expressed in cyanidin-3-glucoside eq./L of juice.

Experimental design and statistical analysis

The optimization of the ultrasonic pretreatment was performed using CCRD. The ultrasound amplitude (x1) and time (x2) were evaluated as independent variables, and response for the total monomeric anthocyanins content was considered. For the experimental design, 12 experiments were carried out in a randomized order and with four repetitions of the central point. The statistical analysis of the data was evaluated by analysis of variance (ANOVA) to verify the mathematical model. The response surface methodology was used to find the pretreatment conditions that provide the highest anthocyanin content in fruit juice. The

STATISTICA (version 7.0) software was used for data treatment.

The Student's t-test was used with a significance level of 5% to evaluate the difference between anthocyanins content of blackberry juices obtained with and without pretreatment with ultrasound.

RESULTS AND DISCUSSION

Optimization of the pretreatment with ultrasound

The experimental design for the ultrasound pretreatment is shown in Table I. The independent variables were time and ultrasound amplitude (min and %, respectively), and the dependent variable was total monomeric anthocyanins content (mg of cyanidin-3-glucoside eq./L of juice). In Table II, the results of the analysis of variance are shown. The coefficient of determination (R^2) for the model was 0.96, and the F value (27.04) was higher than the tabulated value (4.39), which confirms that the

Table I. Experimental design matrix for the pretreatment with ultrasound before juicing by pressing.

| Experiment | Amplitude (%) | Time (min) | TMA* (mg/L) |
|------------|---------------|------------|-------------|
| 1 | 25 | 3 | 388 |
| 2 | 25 | 11 | 373 |
| 3 | 75 | 3 | 440 |
| 4 | 75 | 11 | 484 |
| 5 | 14.6 | 7 | 365 |
| 6 | 85.4 | 7 | 511 |
| 7 | 50 | 1.3 | 437 |
| 8 | 50 | 12.7 | 459 |
| 9 | 50 | 7 | 513 |
| 10 | 50 | 7 | 499 |
| 11 | 50 | 7 | 495 |
| 12 | 50 | 7 | 504 |

*Total monomeric anthocyanins.

mathematical model fits the experimental data. Regarding the model adjustment, it was found that the calculated F value (7.28) was lower than the tabulated value (9.28), which indicates that the lack of fit was insignificant ($p > 0.05$) and the mathematical model is valid.

In Figure 2, it was possible to determine that the conditions for pretreatment with ultrasound changed the content of anthocyanins in the juice. By analyzing the response surface (Figure 2a), it can be seen that the best amplitude and time conditions were found around the central points. For the amplitude of ultrasound, values lower than 50% did not provide an increase in anthocyanins in the juice. However, when an amplitude above 50% is applied, it is possible

to remove higher levels of anthocyanins. When the sonication lasts less than 5 min, this short pretreatment time was not enough to transfer the anthocyanins from the fruit to the juice. On the other hand, for more than 10 min of ultrasound, the content of anthocyanins decreases, probably due to the degradation of these compounds. The degradation of total monomeric anthocyanins due to prolonged exposure to ultrasound is already known, showing that these compounds are sensitive to long periods of extraction with ultrasound (González et al. 2020).

The Pareto chart (Figure 2b) shows the effect of amplitude and time, confirming that both influenced the extraction of anthocyanins into the juice. In addition, the interaction between

Table II. Analysis of variance (ANOVA) for blackberry juices' total monomeric anthocyanins content with the pretreatment with ultrasound.

| Source | Sum of squares | Degrees of freedom | Medium square | F calc | F 0.05 |
|------------------|----------------|--------------------|---------------|--------|--------|
| Regression | 31739.65 | 5 | 6347.93 | 27.04 | 4.39 |
| Residual | 1408.55 | 6 | 234.76 | | |
| Lack of fit | 1238.54 | 3 | 412.85 | 7.28 | 9.28 |
| Pure error | 170.01 | 3 | 56.67 | | |
| Total | 33148.20 | 11 | | | |
| R squared = 0.96 | | | | | |

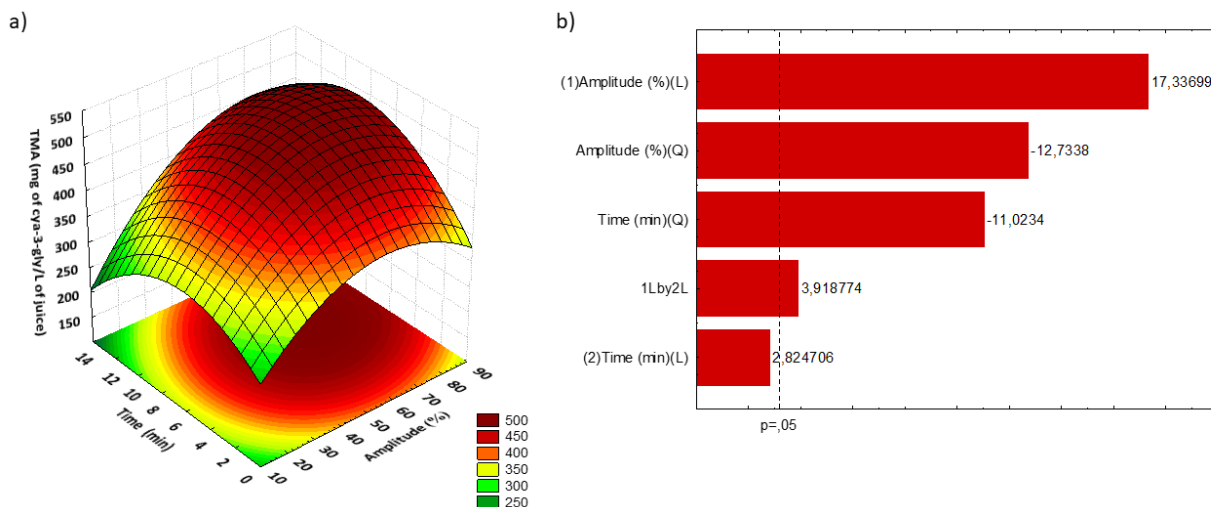


Figure 2. Effect of amplitude and time in the extraction of monomeric anthocyanins from blackberry presented in the forms of response surface graph (a) and Pareto diagram (b).

these parameters also significantly affected the process. Thus, the optimal conditions for pretreatment with ultrasound were those in the center of the response surface graph, using 65% of the ultrasound amplitude for 7 minutes. It is worth noting that these conditions were chosen because they are found in the center of the response surface graph, but if other ultrasound amplitudes higher than 50% were used, the process could provide similar results.

Effect of ultrasound pretreatment on total monomeric anthocyanins content of the juice

When comparing the content of the total monomeric anthocyanins found in blackberry juices with and without ultrasound pretreatment (Figure 3), it can be noted that ultrasound increased total monomeric anthocyanins extraction for the juices of all cultivars, increasing their content by 31% for BRS Xingu and Guarani and 56% for Xavante. Therefore, ultrasound can be used as a pretreatment before pressing to increase the removal of bioactive compounds from the fruit, making the juice more concentrated in total monomeric anthocyanins.

It is well known that the presence of a liquid medium is required for ultrasound extraction of bioactive compounds. The sonication of the solvent leads to the formation of the cavitation effect, in which the generation and implosion of bubbles provide the extravasation of the cellular content of plant tissues (Chen et al. 2020). Since blackberry is composed of nearly 90% water (Moraes et al. 2020), it is possible to expect the cavitation effect without adding water.

Another fact that may have facilitated the extraction was the freezing that the sample was submitted before the pretreatment. Rapid freezing allows the formation of tiny, uniform ice crystals, causing minor damage to cell integrity. Slow freezing, which was used in this study,

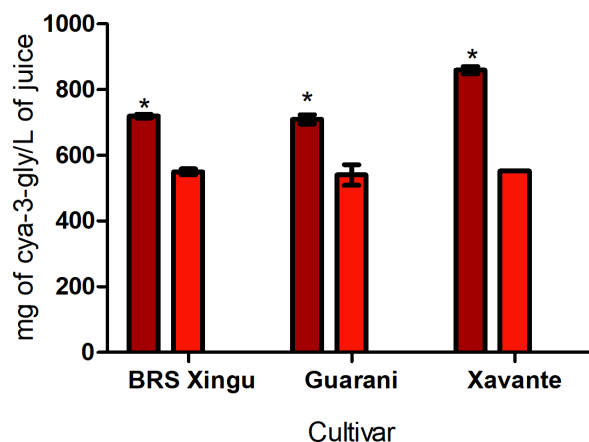


Figure 3. Anthocyanins content of blackberry juices with (strong red) and without (control, weak red) pretreatment with ultrasound. Mean value \pm standard deviation ($n = 3$). The samples that differ using the Student's t-test at a significance level of 5% were identified (*).

produces large ice crystals that cause significant damage to cell structure (Wu et al. 2017). Thus, the damage to the fruit's cellular structure generated by the slow freezing possibly contributed to the ultrasound extraction without the need to add water to the fruits. Hence, it can be seen that the ultrasound waves were transmitted efficiently to the fruits soaked in their own juices and in contact with the flask.

It is important to mention that the implosion of cavitation bubbles generates microjets, which leads to surface flaking, erosion, and particle breakage, facilitating the release of anthocyanins from the intracellular environment to the *in situ* water in the fruit. Sonoporation is another effect reported in ultrasound-assisted extractions, which causes an increase in the permeability of cell membranes and the improvement in the release of cellular content (Chemat et al. 2017). Therefore, sonication can improve extraction efficiency, causing an increase in the mass transfer rate. Thus, through this work it is shown the feasibility of carrying out the pretreatment of whole fruits without adding any solvent to increase the extraction of the fruits,

resulting in juices with a higher concentration of anthocyanins.

Effect of ultrasound pretreatment on the composition of individual anthocyanins in blackberry juices

In Figure 4a and b is shown the anthocyanin profile of blackberry juices without (control) and with ultrasound pretreatment. It can be seen that sonication had a positive effect on increasing the concentration of anthocyanins in the juices. In all cultivars, five anthocyanins were identified, and the same compounds were identified in the ultrasound-treated juices and the controls. Four cyanidins with different ligands were found (cyanidin-3-glucoside, cyanidin-3-rutinoside, cyanidin-3-malonyl-glucoside, and cyanidin-3-dioxyglucoside) (Table III), with

cyanidin-3-glucoside being the major one. Also, pelargonidin-3-glucoside was found in all the studied cultivars.

When evaluating the effect of the pretreatment with ultrasound on the individual anthocyanins present in the juices (Table IV), it is noted that there was a significant increase ($p < 0.05$) of the two major anthocyanins (cyanidin-3-glucoside and cyanidin-3-rutinoside) of blackberries in all cultivars studied. However, the different cultivars showed different behaviors regarding the extraction of each anthocyanin.

As previously reported in the section *Effect of ultrasound pretreatment on anthocyanin content of the juice*, the cultivar Xavante presented the highest content of extracted anthocyanins, where the area of cyanidin-3-glycoside increased by around 53%, and

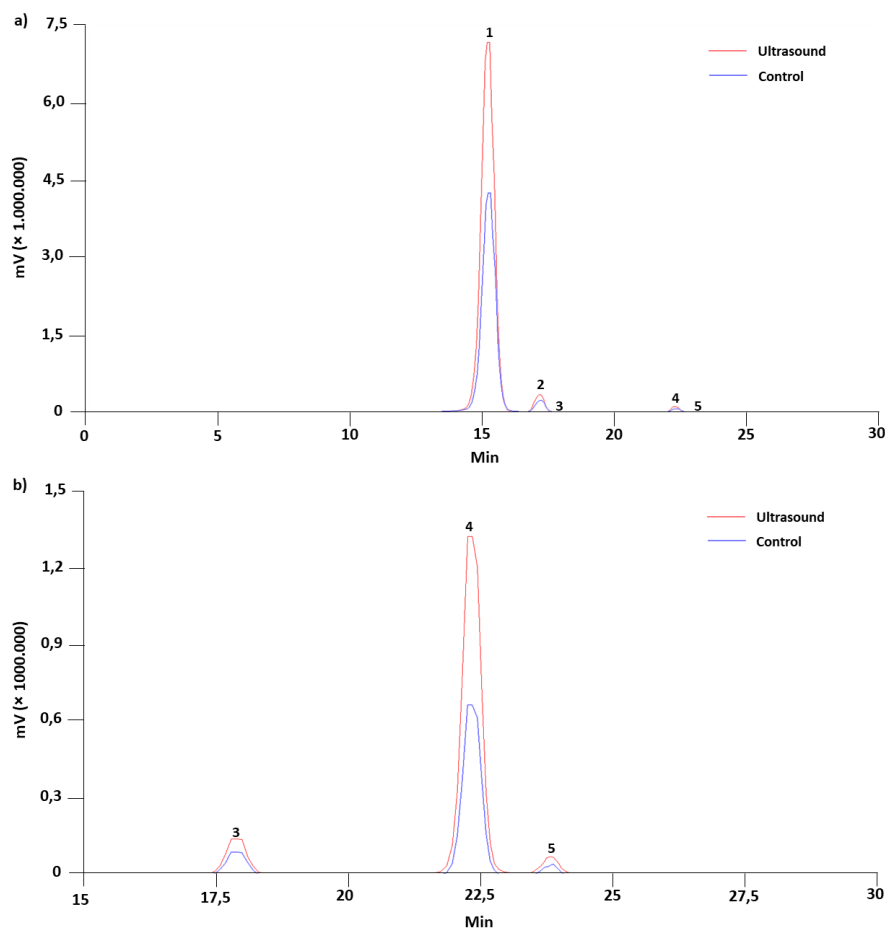


Figure 4. Anthocyanin profile of blackberry juice (Xavante cultivar) without (control) and with pretreatment with ultrasound; (a) complete chromatogram. (b) magnified chromatogram section.

Table III. Anthocyanins identified in the blackberry juices of the BRS Xingu, Guarani, and Xavante cultivars.

| Retention time (min) | Molecular ion and product ion (m/z) | Tentative identification |
|----------------------|-------------------------------------|------------------------------|
| 15.274 | 449-287 | Cyanidin-3-glucoside |
| 17.223 | 595-287 | Cyanidin-3-rutinoside |
| 17.924 | 443-271 | Pelargonidin-3-glucoside |
| 22.391 | 535-287 | Cyanidin-3-malonyl-glucoside |
| 23.863 | 593-287 | Cyanidin-3-dioxalylglucoside |

Table IV. Concentration of individual anthocyanins in blackberry juices.

| Anthocyanin | Cultivar | Treatment | |
|------------------------------|-----------|-----------------------|------------------|
| | | Ultrasound | Control |
| Cyanidin-3-glucoside | | Ultrasound | Control |
| | BRS Xingu | 501 ± 11* (18%) | 424 ± 22 |
| | Guarani | 449 ± 15* (34%) | 334 ± 33 |
| | Xavante | 576 ± 37* (53%) | 376 ± 10 |
| Cyanidin-3-rutinoside | BRS Xingu | 57 ± 4* (110%) | 8.91 ± 1.82 |
| | Guarani | 44.4 ± 0.7* (42%) | 31.3 ± 3.7 |
| | Xavante | 25.3 ± 1.1* (57%) | 16.1 ± 0.1 |
| Pelargonidin-3-glucoside | BRS Xingu | 1.31 ± 0.14 (17%) | 1.12 ± 0.04 |
| | Guarani | 0.7061 ± 0.0002* (7%) | 0.6594 ± 0.00081 |
| | Xavante | 0.96 ± 0.11 (50%) | 0.64 ± 0.07 |
| Cyanidin-3-malonyl-glucoside | BRS Xingu | 8.8 ± 1.2 (12%) | 7.9 ± 0.8 |
| | Guarani | 5.7 ± 0.9 (62%) | 3.5 ± 0.4 |
| | Xavante | 7.8 ± 0.6* (67%) | 4.7 ± 0.6 |
| Cyanidin-3-dioxalylglucoside | BRS Xingu | 18 ± 3 (6%) | 17 ± 2 |
| | Guarani | 3.1 ± 0.1 (5%) | 3.0 ± 0.4 |
| | Xavante | 0.40 ± 0.07 (31%) | 0.31 ± 0.05 |

The results are expressed as mg cyanidin-3-glucoside equivalent per liter of juice. The value in parentheses corresponds to the percentage increase found for each anthocyanin. Mean value ± standard deviation (n = 3). The samples with * differ from each other using the Student's t-test at a significance level of 5%.

cyanidin-3-rutinoside had an increase of 57%. For this cultivar, cyanidin-3-malonyl-glucoside also increased (67% in relation to the control).

Although the BRS Xingu and Guarani cultivars presented the same increase in total monomeric anthocyanins, the individual anthocyanins of each cultivar increased at

different proportions. In the BRS Xingu cultivar, only two anthocyanins differed from the control treatment, increasing 18% for cyanidin-3-glycoside, and 110% for cyanidin-3-rutinoside. As for the Guarani cultivar, the increase was 34% and 42% for cyanidin-3-glycoside and cyanidin-3-rutinoside, respectively. For this same cultivar,

there was only a slight increase of 7% in perlagonidine-3-glycoside.

Thus, it can be seen that each cultivar presented a particular behavior regarding the content of anthocyanin increasing in the juice. However, although the anthocyanins of the three cultivars increased in different proportions, the two main anthocyanins for all samples increased significantly for the juices of sonicated blackberries.

CONCLUSION

Ultrasound can be used as a pretreatment to increase anthocyanins concentration in blackberry juices from 31 to 56% for the varieties studied. The *in situ water* of the fruit was enough to provide ultrasound effects that increased the content of anthocyanins without any solvent. The whole fruit can be sonicated fast and straightforward, allowing a simple reproduction of the experiment or the processing in a higher scale.

Acknowledgments

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brazil (CAPES) - Finance Code 001. The authors thank Embrapa Clima Temperado for the donation of blackberries used in the study.

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How to cite

MORAES DP, FERREIRA DF, FARIAS CAA, NEHRING P, BARCIA MT, CICHOSKI AJ & BARIN JS. 2023. Solvent-free sonication of blackberries for the anthocyanin enrichment of juices obtained by pressing. *An Acad Bras Cienc* 95: e20221106. DOI 10.1590/0001-3765202320221106.

Manuscript received on December 16, 2022; accepted for publication on February 27, 2023

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DPM- Conceptualization, Data curation, Formal analysis, Investigation, Project administration, Methodology, Validation, Writing - Original Draft; DFF; Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Validation, Writing - Review & Editing; PN and CAAF- Formal analysis, Investigation, Visualization, Writing - Review & Editing; AJC- Formal analysis, Investigation, Supervision, Visualization, Writing - Review & Editing; MTB- Methodology, Validation, Project administration, Supervision, Visualization, Writing - Review & Editing; JSB- Conceptualization, Methodology, Project administration, Supervision, Visualization, Writing - Review & Editing.

