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DEVELOPMENT OF FREEZE DRIED SUGARCANE JUICE POWDER

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ABSTRACT: Sugarcane juice is composed by 75 to 82% of water and 18 to 25% of soluble solids, which are constituted by sucrose, glucose, fructose, and lower amounts of amino acids, acids, waxes, fats, pigments, and inorganic salts. Due to its nutritional properties, sugarcane juice has shown potential to fulfill athletes' energy needs. The aim of this work was to obtain sugarcane juice powder using freeze drying, perform the physicochemical characterization of the raw material and the powder obtained and to determine the powder solubility. The sugarcane juice *in natura* showed average soluble solid content of 17.71º Brix and ratio of 46.83; its average contents of calcium and magnesium were 33.94mg.100mL^{-1} and 10.82mg.100mL^{-1}, respectively. The powder showed good solubility and satisfied the legislation requirements with respect to moisture. The water activity obtained (0.49) allowed inhibition of bacterial growth and significant reduction of chemical and enzymatic reactions, contributing to extend the shelf life of products.

Keywords: Sugarcane juice. Freeze drying. Powder. Nutrition.

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1. INTRODUCTION

Sugarcane (*Saccharum officinarum*) belongs to the grass family (Poaceae) and is widely grown in tropical and subtropical regions. Its production results in high socioeconomic benefits due to its high content of sucrose. As raw material, it is characterized as a fresh cut tropical grass that produces culms in advanced state of maturation, wich are usually cut close to the ground and free of strange materials (STUPIELLO, 1987).

In the 2017/2018 season, sugarcane production in Brazil reached 658.8 millions of tons. Among the sugarcane producing regions, Southeast is in the largest producer with 417.47 millions of tons. In the same season, the sugarcane growth area for the production of sugar and ethanol in the country was 8.73 million of hectares (CONAB, 2018).

Sugarcane composition depends fundamentally on factors such as variety, region, climate of the place it is grown, and on its maturity level (HOI; MARTINCIGH, 2013). The plant contains, generally, 65 to 75% of water. Among the solid soluble substances, sucrose is the major component, corresponding to 70 to 91%. Due to its high level of sugar, its protein

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content is extremely low, thus characterizing it as a nutritionally imbalanced food (NOGUEIRA et al., 2009).

Mostly grown for sugar and ethanol production, sugarcane has become a target in the search for the sustainable energy production; since it is a perennial grass, it has high conversion rate from solar energy into biomass, higher than that of other vegetables such as wheat and corn (HOI; MARTINCIGH, 2013). Sugarcane is also used in animal feed and in the production of brown sugar and sugarcane spirit (OLIVEIRA et al., 2012).

Products derived from sugarcane have different biological functions which benefit the human health; therefore it has been considered as anticariogenic and anticarcinogenic. These products have antioxidant effect and reduce progression of atherosclerosis due to the presence of bioactive compounds such as phenols and policosanols (ASIKIN et al., 2014).

Another product derived from sugarcane that is very popular among Brazilian consumers is the sugarcane juice, which is characterized as an energetic, refreshing, non-alcoholic, and nutritive beverage; it is also less expensive than other industrialized beverages (SOCCOL et al., 1990). Obtained by pressing sugarcane stalks, sugarcane juice is composed of 75 to 82% of water and 18 to 25% of soluble solids, among which 15.5 to 24% are the sugars sucrose, glucose and fructose and, in smaller amounts, non-sugars (1 to 2.5%) such as amino acids, acids, waxes, fats, pigments, and inorganic salts (SEGATO et al., 2006).

Like unrefined whole cane sugar, which is a solid form of sucrose derived from the boiling and evaporation of the juice, sugarcane juice is a very energetic food and maintains all sugarcane nutrients, including minerals such as iron, calcium, potassium, sodium, phosphorus, magnesium, and chlorine besides vitamin C and vitamin B complex (FAVA, 2004). It has highly digestible carbohydrates, with energetic values around 661 kcal/kg digestible energy and 637 kcal/kg metabolizable energy (ME) (PRADO et al., 2010).

Due to its nutritional properties, the effectiveness of sugarcane juice in significantly increase athletes' muscular mass has been studied, and the ergogenic effect of the product in the replacement of muscle glycogen in soccer players after training has already been proved (STANCANELLI, 2006).

These characteristics make sugarcane juice a potential food to fulfil the energy needs, especially those of athletes. Therefore, sugarcane juice powder has proven to be an attractive alternative in terms of its ease of consumption.

The powder has several advantages such as maintenance of quality and stability without cooling, which is obtained by water activity reduction leading to the reduction of chemical and enzymatic reactions that occur during food storage. Similarly, value is added to the product and postharvest wastes are reduced (MARQUES, 2009).

The National Health Surveillance Agency (Anvisa) approved the technical regulation (RDC 272, September 22, 2005) on vegetable products, fruit products, and edible mushrooms. According to this Resolution, vegetable products are the products obtained from edible parts of vegetable species traditionally consumed as food and submitted to the drying process, combined or not with other technological processes considered safe for food production, namely dehydration, cooking, salting, fermentation, lamination, flocculation, extrusion, and freezing. As a specific requirement, vegetable products, dried or dehydrated, should have maximum moisture of 12% (BRASIL, 2005).

Dehydration is an old method of food conservation which prolongs shelf life by reducing water activity by inhibiting microbial growth and enzymatic activity. It also has the advantage of reducing product weight, which results in reduction of transport and storage costs (FELLOWS, 2006).

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Among the drying processes used by food industries, freeze drying stands out with high quality products due to the low process temperature and the fast transition between hydrated to dehydrated state, which minimizes many degradation reactions that are common during the drying process, such as Maillard reaction, protein denaturation, and enzymatic reactions (LIAPIS et al., 1985).

The freeze drying technique does not expose substances to high temperatures, which allows the maintenance of their initial nutritional characteristics and the instantaneous return to their original shape and texture (BARUFFALDI; OLIVEIRA., 1998). Although it is characterized as a slow process, it has the advantage of preventing product adherence to the dryer surface, which occurs when heat is involved in the process (SANTO et al., 2013).

Furthermore, an important feature of the separation process by sublimation is the strong structure provided by freezing the material surface where sublimation takes place; it is important to prevent collapses of the solid matrix after drying. As result, it is possible to obtain a pore that is easy to rehydrate after water addition. Although freeze drying does not eliminate microorganisms, they become unable to grow in the dry material (BOSS, 2004).

Therefore, the objectives of this study were to obtain sugarcane juice powder using freeze drying, perform the physicochemical characterization of the raw material and the powder obtained, and to determine the powder solubility.

2. MATERIALS AND METHODS

The sugarcane juice used in this experiment was obtained from a rural property in the city of Lajeado Grande, Santa Catarina state, Brazil, and it was extracted following the Good Manufacturing Practice guidelines. After extraction, the sugarcane juice was sieved through two domestic sieves, transported to the Food Chemistry laboratory of the Food Engineering Department of the Santa Catarina State University, and stored under refrigeration until analyses, which was performed in triplicate. The raw material was characterized by measuring the pH and determining the soluble solids content, titratable acidity, ratio, and content of calcium and magnesium. Next, freeze drying was performed, and the dry material was then crushed manually using a pestle and mortar. The product obtained was analyzed for moisture, water activity, and water solubility index. Titratable acidity and pH were measured again, and the pH was determined at the dilution rate of 1:10 (powder:distilled water). The pH and titratable acidity data of the analyses performed in the sugarcane juice *in natura* and in the sugarcane powder obtained were submitted to ANOVA, and the means were compared using the Tukey Test at 5% level of significance.

2.1 pH

The pH was determined by the potentiometric method using a PHB-500 pH/Ion Meter, previously calibrated with pH 4.0 and pH 7.0 buffers.

2.2 Soluble solids content

Soluble solids content was obtained using a Benchtop Abbe refractometer with direct reading (°Brix). The reading was corrected according to the sample temperature using the correction table proposed by Instituto Adolfo Lutz (INSTITUTO ADOLFO LUTZ, 1985).

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2.3 Titratable acidity

Titratable acidity was determined using 0.1N NaOH. The turning point was determined measuring the pH value (8.1 ± 0.02), according to the AOAC method for colored samples, and the results were expressed as percentage of citric acid.

2.4 Calcium and magnesium determination

The minerals calcium and magnesium were determined using the volumetric method by complexation with EDTA 0.1M solution as chelating agent. Due to their sample organic nature, ashes were obtained by incinerating the sample in a muffle furnace at 550ºC and a sample-solution was obtained. Tha sample-solution was prepared by dissolving the ashes obtained in hydrochloric acid (1:1), 2 drops of nitric acid, and distilled water. The resulting sample was then filtered using a small funnel and the filtrate was collected in a 100 mL volumetric flask, completing the final volume with distilled water (Instituto Adolfo Lutz, 1985).

For the determination of calcium, titration was performed using 0.1% Calcon indicator until change in the color of solution, which indicated the equivalence point. The results were expressed as mg of calcium per 100mL of sample.

A new titration was performed substituting the Calcon indicator for Eriochrome black T 0.1% for magnesium determination. The calcium content obtained was subtracted from the calcium and magnesium content obtained in this titration; the result was expressed as mg of magnesium per 100mL of sample.

2.5 Freeze drying

The samples were previously frozen at -86ºC in an Indrel ultra-freezer (IULT) for 40 minutes. Then, they were dried using a freeze dryer (IlShin - TFD 5503) for 25 hours, when it was observed reduction by more than 80% in the initial weight of the samples, value related to the product moisture (SEGATO et al., 2006).

2.6 Moisture

The sugarcane juice powder moisture was obtained by the method proposed by Instituto Adolfo Lutz for sugars and correlated products using a stove at 105 ± 2ºC until constant weight (INSTITUTO ADOLFO LUTZ, 1985).

2.7 Water activity

The water activity of the sugarcane juice powder was determined using a Pro Analysis Testo 650 measuring instrument by inserting the sample and waiting until the instrument stabilized.

2.8 Water solubility index

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Water solubility index was determined based on the method by Astolfi-Filho et al. (2005), who determined passion fruit juice solubility measuring the time needed to complete dissolution of 10g of the product in 100mL of distilled water. The mixture was manually shaken using a glass stick, and the time, measured using a chronometer, corresponded to the moment when solid particles were no longer observed.

3. RESULTS AND DISCUSSIONS

The average results obtained in the physicochemical characterization of the sugarcane juice *in natura* and sugarcane powder are shown in Tables 1 and 2, respectively; all analyses were performed in triplicate.

Table 1- Physicochemical determinations in the sugarcane juice *in natura*

Sugarcane juice *in natura*	Mean ± standard deviation
pH	5.45 ± 0.05
Soluble solids content (ºBrix)	17.71 ± 0.24
Titratable acidity (% citric acid)	0.38 ± 0.05
Ratio (ºBrix/acidity)	46.83 ± 0.64
Calcium ($mg.100mL^{-1}$)	33.94 ± 0.05
Magnesium ($mg.100mL^{-1}$)	10.82 ± 0.65

Source: authored by the authors (2018).

Table 2- Physicochemical determinations in the sugarcane juice powder.

Sugarcane juice powder	Mean ± standard deviation
pH	5.53 ± 0.08
Titratable acidity (% citric acid)	12.99 ± 1.67
Water activity	0.49 ± 0.00
Moisture (%)	6.97 ± 1.17
Solubilization time (s)	50 ± 1.41

Source: authored by the authors (2018).

The pH value of 5.45 found in the sugarcane juice *in natura*, as presented in Table 1, is close to the values obtained by Oliveira et al. (2007), pH of 5.28, Silva and Faria. (2006), pH of 5.3, and Marques (2009), pH of 5.46. The pH of the sugarcane powder, shown in Table 2, was 5.53 and it was not significantly different ($p<0.05$) from the initial value. Since the acids present in sugarcane are organic acids, which are considered weak acids, even in high amounts, they are not sufficient to reduce the pH of sugarcane juice, which leads to small pH variations between different experiments (MARQUES, 2009).

As for the titratable acidity value that can be seen in Table 1, 0.38% of citric acid in the first measurement, it is close to the value obtained by Marques (2009), 0.44%, while Oliveira et al. (2007) and Silva and Faria (2006) found 0.04% and 0.056%, respectively. Kunitake (2012) attributed the differences observed in the sugarcane juice composition to the variations in the vegetable composition due to different planting periods, harvest time, climate conditions, and maturation stage. Kunitake (2012) investigated nine batches of sugarcane from the same cultivars and same source of production for approximately 12 months and

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obtained titratable acidity values varying from 0.038 to 0.071% of citric acid. The differences between the values of titratable acidity obtained by different authors can be explained by the differences between growing region, cultivar, and climate of the sugarcane used as raw material in the different experiments. Due to degradation reactions caused by microbial activity, before or after sugarcane processing, other acids can be formed, such as acetic, lactic, and formic acids. Therefore, acidity variations can result from bacterial activity (WOJTCZAK et al., 2013).

Table 2 shows the total acidity of the dry product which increased reaching the value of 12.99% showing a significant difference in relation to the total acidity value of sugarcane juice *in natura*. Moreira et al. (2013) observed difference of 0.47% of citric acid in mango pulp *in natura* and 3.28% of citric acid in the freeze dried powder product, confirming the increase in the acidity level due to the concentration of organic acids during the drying process. The increased acidity of the powdered product ensure its characteristic acidic flavor when it is reconstituted or used as a food supplement, besides preventing microbial growth (OLIVEIRA et al., 2011).

The soluble solids content obtained was 17.71º Brix (Table 1). Silva and Faria (2006) found content of 19º Brix, Marques (2009) obtained 18.24º Brix, and Oliveira et al. (2007), 22.74º Brix. Tasso Junior et al. (2007) found values varying between 18.64 and 23.1 and Santos et al. (2011) obtained values between 20.25 and 23.30, who believe that the soluble solids content of sugarcane juice is strongly influenced by the fertilization of the soil where the plant is grown. Among the soluble solids present in sugarcane juice, sugars stand out, with sucrose around 17%, glucose, 0.4%, and fructose, 0.2% (OLIVEIRA et al., 2007).

Table 1 also presents the ºBrix/acidity ratio obtained in the present study, 46.83. The juice industry usually uses this parameter to determine product sweetness, allowing the standardization of the juice for processing based on this value (SHIGEOKA, 1999).

The contents of calcium and magnesium in the sugarcane juice *in natura* are presented in Table 1 and equal to 33.94mg.100mL^{-1} and 10.82mg.100mL^{-1} of sample, respectively, similar to those obtained by Oliveira et al. (2007), 31mg.100mL^{-1} of calcium and 10mg.100mL^{-1} of magnesium. Nogueira et al. (2009) obtained values between 14 and 48mg.100mL^{-1} of calcium and between 12 and 25mg.100mL^{-1} of magnesium. Neves (2004) found calcium contents between 9.6 and 15.12mg.100mL^{-1} in sugarcane juice, investigating different varieties of sugarcane.

The moisture content of the sugarcane juice powder was 6.97%, shown in Table 2, which meets the current requirements established by Brazilian law. Righetto (2003) obtained moisture content of 10.79% for freeze dried pure acerola juice, and Moreira et al. (2013) found moisture of 3.14% for freeze dried mango pulp powder; both values are related to their drying time.

Water activity refers to the degree of availability of water in a food and is defined as the relation between the fugacity of water in the food and the fugacity of pure water at the same temperature (DITCHFIELD, 2000). The water activity value obtained in the present study, 0.49 at 22.5ºC, was higher than that found by Marques (2009), 0.360 at 25ºC in sugarcane juice powder obtained by foam mat drying. These results show that water activity may be related to the food drying method. Studying freeze drying in tropical fruits, Marques (2008) found differences in the water activity values according to the different freezing techniques used, which leads to different cell structures and products with distinct porous structures. Thus, Marques (2008) considered the use of freezing with nitrogen in liquid and vapor states more efficient and obtained water activity values of 0.22 and 0.19, respectively. In the water

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activity range from 0.4 to 0.6 (the results found in the present study fall in this range), there is a drastic reduction in the enzymatic activity and hydrolytic reactions. The growth of fungi, yeast, and bacteria is inhibited influencing product conservation (DITCHFIELD, 2000).

Table 2 shows the solubility of the sugarcane juice powder, determined as the time needed to complete dissolution of 10g of the product in 100g of distilled water at 25ºC, which was 50s. Astolfi-Filho et al. (2005) found times for solubilization in microencapsulated passion fruit juice varying between 36.71 and 60s, and the time increased with an increase in the proportion of juice added. Time for solubilization up to 60s is considered characteristic of products with good solubility. In the case of sugarcane juice, this factor can be influenced by the presence of high levels of sucrose, a very soluble compound (ASTOLFI-FILHO et al., 2005).

4. CONCLUSION

The sugarcane juice *in natura* showed average soluble solid content of 17.71º Brix and ratio of 46.83; its average contents of calcium and magnesium were $33.94 mg.100mL^{-1}$ and $10.82 mg.100mL^{-1}$, respectively.

Freeze drying resulted in a sugarcane juice powder with good solubility. An easy rehydration by water addition is a characteristic of the process. The high quality of the product obtained by freeze drying results from the use of low temperature, which prevents chemical and enzymatic reactions.

The sugarcane juice powder obtained meets the current legislation standards for vegetable products, with moisture value of 6.97%. The value of water activity obtained, 0.49, and the increase in the final product acidity, 12.99%, are conditions that are not suitable for the growth of microorganisms, which facilitates the product conservation.

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