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Production of Mango and Passion Fruit Mix Spirit in a Distillation Column

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Cachaça is the most consumed distilled beverage among Brazilians and the third in world consumption. In order to innovate and expand the spirits chain, new technologies have been developed. This work aimed to evaluate the development process of a distilled beverage from the mixture of mango and passion fruit pulps, using a batch distillation column. The column has a collection system with seven fractionation modules, jacketed, with fillings, vacuum thermal insulation and temperature monitoring. For the production, the wort was prepared from the same proportions of the fruit pulps and led to the fermentation, where the kinetic monitoring was carried out until the wine is produced. Thereafter, the wine was distilled in two serial batches of 4.5 L of drink, in a controlled process in order to obtain a quality brandy through the proper separation of the head, heart (commercial) and tail fractions. The fractions were characterized and the "heart", marketed product, compared to parameters determined by the Brazilian legislation for the composition of fruit spirits. It was noticed that the distillate complies the legislation, being its consumption released without risks to health. It is noteworthy that the spirit obtained from the fruit mixture proved to be a diversified product within the alcoholic beverages sector, and there were no similar studies to compare the results.

1. Introduction

Among the alcoholic beverages, the cachaça, or sugarcane spirit, has great economic, cultural and social importance for Brazil, where Brazilian customs are intertwined with the origin of the drink, making it among the most consumed in the country (Alcarde, 2014). Fruit spirits are a new alternative, both by the addition of differentiated sensorial characteristics as for contributing to the reduction of crop surpluses. Increase the productivity, harnessing inputs and improving products to greater value aggregation are some of the goals associated with technological innovation, a challenge to change usual characteristics and to cause impacts in conditions of competition, investment and profitability (Raimundo et al., 2017).

Fruit production is responsible for the income of many countries, and in Brazil, several varieties of fruits have sensorial qualities that are highly coveted worldwide. Because they are not only consumed *in nature*, due to the high perishability and losses of the fruits, the pulp has important application as raw material for the industry, being able to be stored and processed at any time of the year, becoming an incentive to the incorporation in several processes, such as fermentation and distillation, already existent technologies because they are efficient and low cost (Alonso et al. 2015; Silva et al., 2015).

Mangoes (*Mangifera indica* L.) are the most popular tropical fruits in the world, due to high nutritional value and exotic characteristics, economic importance in national and international markets. The Tommy Atkins variety is responsible for most of the plantations due to its resistance to pests and diseases (Oliveira et al., 2018). Passion fruit (*Passiflora edulis*) is native to tropical America and widely cultivated in Brazil. The fruits, after harvesting, become highly perishable, due to intense respiratory activity and significant loss of water, which leads to the occurrence of changes in fruit appearance, the main parameter of choice among

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consumers. The passion fruit has greater economic importance in industrialized products that use it as raw material by the excellent sensorial acceptance (Rodrigues et al., 2015).

Until obtaining the sprit, the process goes through two major stages, fermentation and distillation, which influence the formation and concentration of chemical compounds and, consequently, the quality of the beverage. The wort is fermented to convert the substrate into ethanol (C_2H_5OH) and carbon dioxide (CO_2) by the action of selected yeasts, and then the so-called fruit wine is distilled to separate the volatile components and increase alcohol concentration. During distillation, the separation of "head", "heart" and "tail" fractions are required, with the "heart" fraction being the marketable beverage, which will either continue to age in barrels or be led to direct storage. According to Alcarde (2014), the ideal cut point of the fractions results in a safe and quality drink. The distillation process can occur in copper stills, usually used in artisanal processes, and in columns, in industrial processes. The distillation columns are towers composed of perforated plates that allow heat exchange between the liquid and vapor phases during the process. They are used in large-scale productions, in continuous processes, where wine intake and distillate and vinasse exits occur in parallel (Ferreira et al., 2018). According to Rodríguez-Bencomo et al. (2016), column distillates have lower amounts of harmful compounds, such as acetaldehyde and methanol, as well as higher alcohols, alcohols and higher esters when compared to stills. This work develops a mixed spirit of mango and passion fruit pulps in a distillation column, in order to branch out the alcoholic beverage chain, evaluating the entire production process.

2. Materials and Methods

The fruits selected were washed and sanitized. Then, they are depulped and distributed in plastic containers for storage in freezer until processing.

2.1 Preparation and characterization of alcoholic fermentation

Fermentation was formulated in the pre-studied ratio of 1:4 (pulp and water), where the pulp part was divided into equal fractions of mango and passion fruit. The obtained broth was filtered, chaptalized (added sugar into must) to 18 °Brix and adjusted to pH 4.5 with 20% (w/v) sodium carbonate solution (Na_2CO_3). It was added 0.1 g/L of magnesium sulphate ($MgSO_4$) and 1 g/L of ammonium phosphate ($NH_4H_2PO_4$), supplements to the fermentation medium. The wort was pasteurized at 60°C for 30 min and, after cooling in an ice bath to ambient temperature it was added 0.1 g/L of sodium metabisulfite ($Na_2S_2O_5$) to prevent oxidation of the must.

The inoculum used for the fermentation was a commercial *Saccharomyces cerevisiae* yeast, in the form of a dry yeast, at the concentration of 10 g/L. 20% (v/v) of the wort was used to prepare a pre-inoculum, and after 24 h it was added to the rest of the wort. The fermentation was conducted in 20 L bottle, adapted with a hose to release CO_2. Daily aliquots were collected to follow the fermentation, and the end of the process was given by constant reading of the soluble solids content (ºBrix). At the end, the bottle was stored under refrigeration for yeast sedimentation, withdrawal of the broth and beginning of the distillation.

The kinetics of alcoholic fermentation was analyzed for total soluble solids, total reducing sugars (TRS), cell growth and product formation(ethanol). Cellular growth monitoring (X) was performed using the absorbance measurement in the LGI-VS-721N spectrophotometer, at 600 nm, with a standard curve relating dry weight and absorbance. The concentration of substrate (S) was determined by the colorimetric method of 3.5-dinitrosalicylic acid (DNS), after hydrolysis of the broth with H_2SO_4 1.5 M and neutralization with NaOH 2 N. The ethanol content was determined by simple distillation and spectrophotometry after reaction with potassium dichromate (Joslyn, 1970). The soluble solids content (ºBrix) was measure by digital refractometer Hanna HI96801.

From the data obtained it was evaluated the cells yield ($Y_{X/S}$, g/g), the ethanol yield ($Y_{P/S}$, g/g), the productivity in ethanol (PE g/Lh), and the maximum specific growth rate ($\mu_{máx}$, h^{-1}).

2.2 Distillation of fermented and obtained spirit

The distilled beverage was obtained in distillation column with thermal insulation under vacuum, temperature monitoring and sample collection system with seven glass/Teflon modules and seven jacketed fractionation modules containing Rasching ring filling. The process was conducted in batch mode, with 4.5 L each. At the end of the process, the fractions from each batch were homogenized.

The fractions "head", "heart" and "tail" were separated according to the alcohol content. Distillation started, 25 mL samples were collected from the product sample collector to analyse the alcohol content using a portable digital densimeter DA-130 (Kyoto Eletronics). Samples collected with values above to 60% of alcohol content constituted the "head" fraction; between 60 and 29%, the fraction "heart", and from 29% to 3%, the "tail" fraction.

2.3 Characterization of the distillate

It was possible to evaluate the fractions regarding the limits of the Brazilian legislation for fruit spirits following the methodologies of Alcarde et al. (2012) and Bortoletto and Alcarde (2013). The distillate was analyzed for ethanol, volatile acidity, aldehydes, esters, methyl alcohol, 2-butanol alcohol, isobutanol, 1-butanol, isoamyl alcohol, superior alcohols, furfural and coefficient of congeners.

Aliquots of 1.0 μL were automatically injected into the gas chromatographic system (Shimadzu QP-2010 PLUS, Tokyo, Japan) equipped with a Stabilwax-DA capillary column (Crossbond carbowax polyethylene glycol, 30 m × 0.25 mm × 0.25 μm film thickness) and a flame ionisation detector (FID). The entrainment gas was N_2, with flow of 1.2 mL/min. The temperatures of both the injector and the detector were set at 220 °C. The oven temperature program was 35 °C for 5 min, followed by an increase to 220 °C at a rate of 4 °C/min, with retention of 10 min at 220°C.

3. Results and Discussion

3.1 Fermentation kinetics

The soluble solids content (ºBrix) was used as the main parameter for the end of the fermentation. The broth started with a value of 18.1 ± 0.10 °Brix, and remained constant at 5.7 ± 0.06 °Brix, with 7 days fermentation process. The pH of the medium dropped from 4.5 on the second day to 4.0 and remained stable to the end, as shown in Figure 1a. In order to evaluate the whole fermentative process, the kinetics of cell growth (X), substrate consumption (S) and product formation (P) were monitored during fermentation (Figure 1b).

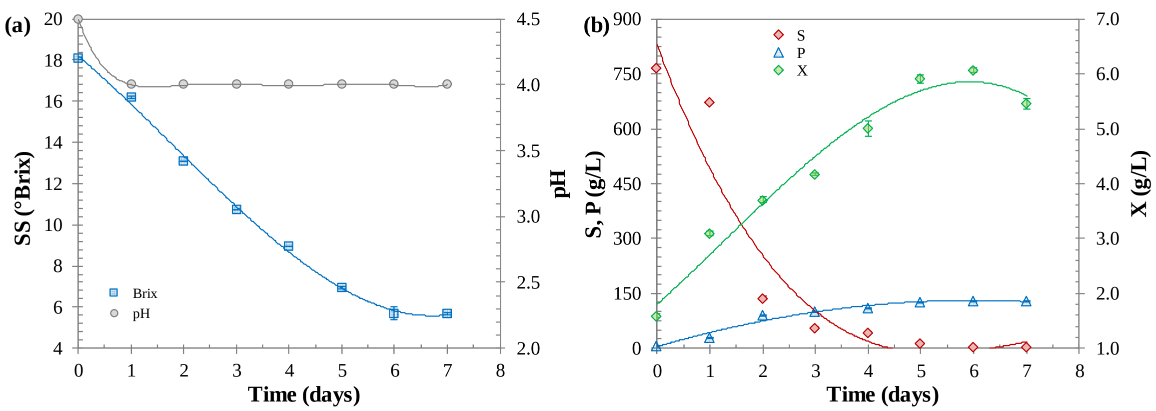

Figure 1: Fermentation kinetics of the fermented mango. a) Soluble solids (Brix) and pH; b) Substrate consumption (S), growth rate (X) and ethanol production (P)

The rapid cellular growth and easy adaptation of the yeasts, with high consumption of the substrate, especially in the first days of fermentation, may be related to the pre-inoculum. It is also possible to visualize a relationship between the profiles at the end of the process, in which the growth rate is determined by the decline and the substrate consumption and the formation of the product stabilize, similar to the soluble solids content, finishing the process.

In the general evaluation of the process, the cells yield ($Y_{X/S}$) of 0.02 g/g, was close to that obtained by Nascimento et al. (2018) in the fermentation in residual broth of the jabuticaba bark ($Y_{X/S}$ = 0.028 g/g). The ethanol yield ($Y_{P/S}$) of 0.67 g/g was lower than those reported by Alvarenga et al. (2013) in mango (0.83) and banana (0.77). A productivity of ethanol (P_E) of 1 g/Lh was a value within the range obtained in the alcoholic fermentations of different treatments of kiwi juice, 0.74 to 2 g/Lh, obtained by Bortoloni et al. (2001).

The maximum specific rate of cell growth (μ_{max} = 0.04 h^{-1}) was low in relation to the fermented palm fruit produced by Lopes et al. (2005), 0.16 h^{-1}. This parameter is influenced by factors such as the yeast strain used, fermentation conduction, temperature and substrate (Almeida et al., 2006).

3.2 Characterization of the distillate

Table 2 shows the results of the chromatographic analyzes of the fractions of the distillate, together with limits established in Brazilian law (Normative Instruction nº 15/2011, Attachment VI) for fruit spirits, which must have an alcoholic content between thirty-six to fifty-four percent by volume, at 20 °C, and be obtained from simple alcoholic distillate of fruit or from the distillation of fermented grape must (Brazil, 2011).

The alcohol content obtained in the spirit of the mix was 48.71ºGL, close to those found for carambola spirit by Moreira et al. (2018) and in the production of spirit from cassava starch residue by Vilhalva et al. (2013) of 43ºGL.

Table 1: Chemical composition of distillate fractions and MAP quality parameters for fruit spirits

	Brazilian law for fruit spirit	Head	Heart	Tail
Alcoholic strength at 20ºC (v/v)	36-54	85.54	48.71	5.81
Volatile acidity in acetic acid*	0-150	8.57	11.23	84.17
Aldehydes in acetic aldehyde*	max 30	19.49	6.92	nd
Esters in ethyl acetate*	max 250	138.08	0.02	nd
Methyl alcohol*	<20	1.62	8.66	25.99
2-butanol *	0-10	0.25	nd	nd
1-Propanol*	-	32.89	18.64	25.30
Isobutanol*	-	154.79	40.07	17.56
1-Butanol*	0-3	1.59	0.88	nd
Isoamyl alcohol*	-	517.07	240.87	10.67
Superior alcohols*	max 360	704.75	299.59	53.53
Furfural*	max 5	nd	nd	nd
Coefficient of congeners*	200-650	870.88	317.76	137.69

nd = not detected; * mg/100 mL of anhydrous alcohol

The volatile acidity is related to the asepsis of the process, being high values an indicative of contamination or erroneous collection of the fractions during the distillation, and low values a quality product (Gonçalves et al., 2009). For spirits mix, the volatile acid value was 11 mg/100 mL of anhydrous alcohol. Da Silva et al. (2009), in the study of the physical-chemical quality of banana pulp and whole banana spirits, identified the values of 8.06 mg/100 mL of anhydrous alcohol for the whole banana and 24.4 mg/100 mL for the banana pulp.

Acetic aldehyde is the main aldehyde present in the distillates and an indicator of ethanol oxidation during the fermentation process, where its presence characterizes low quality of the spirit. At high concentrations, it has strong, cloying taste (Parazzi et al., 2008; Barcelos et al., 2007).

For ethyl acetate, the results were compared with the grape, cane and orange spirits produced by Cleto and Mutton (2004), which had values of 34.7 mg/100 mL of anhydrous alcohol (AA), 33.7 mg/100 mL of AA and 37.9 mg/100 mL of AA, respectively, observing that the higher the acidity in the fermentation broth, the higher the ethyl acetate value present in the spirit. In this study, a much lower value (0.02 mg/100 mL of AA) was found in the consumable fraction (“heart”) and a considerably high value in the “head” (138.08 mg/100 mL of AA), suggesting that this component has a higher volatility and the sampling should be controlled to separate it correctly.

The concentration of 62 mg of methanol per 100 mL of AA was found by Alves et al. (2008), in the development of guava spirit, much higher than that found here, even for the other unused fractions. Methanol is harmful to humans and, if consumed in high concentrations, affects the respiratory system and, in some cases, can lead to death (Almeida et al., 2006).

The superior alcohols were obtained by the sum of the n-propyl, isobutyl and isoamyl alcohols, and it is noted that the latter is responsible for the greater value of the sum. According to Barcelos et al. (2007), superior alcohols are metabolic products and depend on the conditions of the medium, inoculum, temperature, equipment and distillation methods.

In none of the fractions it was detected the presence of furfural. According to Boza and Oetterer (1999) apud Asquieri et al. (2009), the degradation of the sugar still present in the wine with the prolonged heating of the distillation can be a way to give the furfural, where when formed it can attribute fiery flavor.

Coefficient of congeners is the sum of the “non-alcohol” volatile components, such as volatile acidity, aldehydes, esters, higher alcohols and furfural. When these separate compounds have high concentrations, consequently, the congener coefficient will also be (Parazzi et al., 2008). The distillation "head" fraction presented the highest value, due to the concentration of the superior alcohols and esters have significant values.

4. Conclusions

The union of mango and passion fruit pulps proved to be a technologically viable alternative for the production of spirits, since, in addition to adding value to the fruits, it complies with current legislation without presenting toxic compounds that are harmful to health. This also represents the efficiency of the distiller in the column of dishes in the fractionation and obtaining of the distillate. The lack of lines of work as it stands, despite the difficulty in comparing the results, enables the introduction of a new and differentiated product in the market. It is now part of the assessment of the beverage produced, in the face of aging and consumer acceptance.

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