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Characterization of different fruit wines made from cacao, cupuassu, gabiroba, jaboticaba and umbu

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ABSTRACT

The main aim of this work was to produce fruit wines from pulp of gabiroba, cacao, umbu, cupuassu and jaboticaba and characterize them using gas chromatography–mass spectrometry for determination of minor compounds and gas chromatography-flame ionization detection for major compounds. Ninety-nine compounds (C_6 compounds, alcohols, monoterpenic alcohols, monoterpenic oxides, ethyl esters, acetates, volatile phenols, acids, carbonyl compounds, sulfur compounds and sugars) were identified in fruit wines. The typical composition for each fruit wine was evidenced by principal component analysis and Tukey test. The yeast UFLA CA 1162 was efficient in the fermentation of the fruit pulp used in this work. The identification and quantification of the compounds allowed a good characterization of the fruit wines is a viable alternative that allows the use of harvest surpluses and other underused fruits, resulting in the introduction of new products into the market.

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1. Introduction

There is an abundance of exotic tropical fruits in Brazil with the potential to be used by the food industry. Different new uses and new methods for processing tropical fruits need to be developed to minimize production losses, generate more profits and promote the sustainable use of biomes, such as the *cerrado* (Brazilian savannah) and the Amazon forest. One possible use of these fruits is in the production of fruit wines (Dias, Schwan, Freire, & Serôdio, 2007; Duarte, Dias, Pereira, Gervásio, & Schwan, 2009).

There are many studies in the literature that demonstrate the feasibility of using fruits, such as cacao (Dias et al., 2007), gabiroba (Duarte et al., 2009), kiwi (Soufleros et al., 2001), cajá (Dias, Schwan, & Lima, 2003), mango (Reddy & Reddy, 2005) and orange (Selli et al., 2008) to produce alcoholic beverages.

There are several Brazilian fruits with the potential for use in the production of wines. In this study, we investigated the following fruits for this purpose: cupuassu (*Theobroma grandiflorum* Schum.), umbu (*Spondias tuberosa* L.), gabiroba [*Campomanesia pubescens*

(DC.) O. Berg], cacao (*Theobroma cacao* L.) and jaboticaba (*Myrciaria jaboticaba* Berg).

Cupuassu is a fruit native to the Brazilian states of Maranhão and Pará and is one of the most consumed fruits in that region. Some authors consider cupuassu as one of the most promising fruits for commercialization among many others of the Amazon region (Quijano & Pino, 2007). The cupuassu pulp has an average pH of 3.4 and its sugar content is about 10.7 °Brix. It is used to produce juice, ice cream, jams, liqueur, filling for chocolates, and other products. Umbu is a fruit native to the semi-arid regions in the Brazilian northeast. It is consumed locally as fresh fruit, in juices and as ice cream. Umbu pulp has a pH of 2.2 and a sugar content of 14.8 °Brix; these values may vary according the climate of the region of origin of the plant (Lira Júnior et al., 2005). Gabiroba is a fruit native to the western and southern Brazilian savannah. This fruit has been rated as a potential food source for both domestic fowl and humans. Gabiroba is consumed fresh locally and is also used in the production of homemade ice cream, jams, juices and sweets. The pulp of the gabiroba has a pH of 4.1 and a sugar content of about 14 °Brix; these values, combined with good pulp yields, allow for the use of gabiroba fruits in wine production (Duarte et al., 2009). Cacao is known worldwide for its beans, which are used in the production of chocolate. The production and commercialization of

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cacao beans have long been the basis of the economy of some Brazilian states, especially Bahia (Dias et al., 2007). The pulp of the cacao fruit is a substrate rich in nutrients; it is a by-product of the processing of the fruit and can be used in the production of wines and other products (Schwan & Wheals, 2004). The jaboticaba tree, also known as the "Brazilian grape tree", is a tree native to Brazil that belongs to the Myrtaceae family. Its fruits are purplish black, and their skin and pulp have a sweet taste and low acidity. Jaboticaba fruits are consumed fresh and in processed forms such as jams, juices and liqueurs.

Alcoholic fermentation leads to a series of by-products in addition to ethanol. They include carbonyl compounds, alcohols, esters, acids and acetals, all of them influencing the quality of the finished product. The composition and concentration levels of the by-products can vary widely $(ngL^{-1} to hundreds of mgL^{-1})$ (Plutowska & Wardencki, 2008). Although the number of publications about fruit wines has increased in recent years the chemical characterization of these beverages has not been detailed. The purpose of this study was to elaborate alcoholic beverages from cacao, cupuassu, gabiroba, jaboticaba and umbu pulps and characterize them using gas chromatography-mass spectrometry (GC-MS) for determination of minor compounds and gas chromatography-flame ionization detection (GC-FID) for major compounds. Additionally, glycerol, ethanol, sugars and organics acids were also detected by high-performance liquid chromatography (HPLC). It is expected that the determination of the compositions of these beverages will allow for better use of these fruits in the production of fruit wines.

2. Materials and methods

2.1. Must preparation

The fruit wines made from the selected fruits were prepared according to Dias et al. (2007) and Duarte et al. (2009). The fruits of gabiroba, jaboticaba, umbu, cupuassu and cacao were harvested between September and December 2008 and 10 kg of each fruit were selected, washed and mechanically depulped for the must preparation. The fruit pulps were diluted with a sucrose solution to adjust the sugar content to 16 °Brix, and the pH was adjusted to 4.5 with the addition of calcium carbonate. Hydrolases were added to facilitate juice clarification and an enzyme solution with polygalacturonase and cellulase activities (Ultrazym AFP-L, Novozymes, Novo Nordisk Ferment Ltd, Fuglebakken, Denmark, 100 Units mL⁻¹) was added to a concentration of 0.7 mLL⁻¹. Sulfur dioxide, in the form of potassium metabisulfite, was added up to a concentration of 100 mg L⁻¹ of free SO₂ to inhibit bacterial growth. Also, bentonite was added (10 gL^{-1}) to the must to facilitate the sedimentation of non-fermentable solids. The bentonite had been previously suspended in water to a concentration of 10 gL^{-1} to aid its dispersion in the must.

2.2. Fermentation assays

Six fermentations were performed: five of them (cacao, cupuassu, gabiroba (I), jaboticaba and umbu) were inoculated with 10^8 cells mL⁻¹ of *Saccharomyces cerevisiae* UFLA CA 1162 and the other one (gabiroba (NI)) was allowed to ferment spontaneously with the gabiroba pulp. All vinifications were carried out in 5 L flasks in a cold room at 22 °C and the fermentation was monitored by the daily measurement of Brix value, CO₂ and temperature. The fermentation was considered complete when the Brix level was stable. At the end of fermentation, the vats were transferred to a 10 °C incubator to aid the sedimentation of solid material from the fruits pulp. After 10 days at this temperature, the wine transfer

was carried out with some aeration and the beverages were incubated at 10 °C for another 30 days. After that period, another transfer without aeration was carried out and the fruit wines were left for another 10 days at 10 °C, prior to filtration (Dias et al., 2007). The fruit wines were then filtered using cellulose filters and stored at 10 °C in glass bottles fully filled to avoid oxygen entrance. All assays were carried out in triplicate.

2.3. Analytical methods

2.3.1. Chemicals

1-Hexanol, (E)-3-hexen-1-ol, (Z)-3-hexen-1-ol, (E)-2-hexenol, 2-pentanol, 3-methyl-3-butene-1-ol, 4-methyl-1-pentanol, 2-heptanol, 3-methyl-2-buten-1-ol, 3-methyl-1-pentanol, 3-ethoxy-1propanol, 1-heptanol, ethyl propionate, ethyl butyrate, ethyl 2methylbutanoate, ethyl 3-methylbutanoate, ethyl hexanoate, ethyl pyruvate, ethyl lactate, ethyl octanoate, ethyl 3-hydroxybutanoate, diethyl malonate, ethyl 2-furoate, diethyl succinate, diethyl glutarate, diethyl malate, mono-ethyl succinate, triethyl citrate, propyl acetate, linalool, myrtenol, methyl salicylate, 4-vinylguaiacol, vanillin, 3,4,5-trimethoxyphenol, propanoic acid, 2-methyl butyric acid, 3-methyl butyric acid, heptanoic acid, octanoic acid, octanal, 6-methyl-5-hepten-2-one, nonanal, 3-(methylthio)-1-propanol, benzothiazole, N-(2-phenylethyl)acetamide, tyrosol, tetradecanoic acid, methanol, 2-phenylethanol, malic acid were purchased from Aldrich Chemistry (Munich, Germany). 1-Butanol, 1-pentanol, 2ethyl-1-hexanol, 1-octanol, furfurol, 1-phenylethanol, ethylphenyl acetate. 2-phenylethyl acetate. 2-methylpropyl acetate. (E)-furan linalool oxide, (Z)-furan linalool oxide, (E)-pyran linalool oxide, (Z)pyran linalool oxide, geranic acid, isobutyric acid, butyric acid, hexanoic acid, nonanoic acid, octanoic acid, hexadecanoic acid, 3hydroxy-2-butanone, 2-furaldehyde, 2-phenoxyethanol, acetaldehyde, 1,1-diethoxyethane, 1-propanol, 2-methyl-1-propanol, 2-methyl-1-butanol, 3-methyl-1-butanol were purchased from Fluka Analyticals (Seelze, Germany). Limentol, linalool hydrate, αterpineol, 4-terpineol, ho-trienol, borneol, citronellol, geraniol, verbenone, δ -decalactone were purchased from Lluch (Barcelona, Spain). Menthol, benzyl alcohol, ethyl acetate, succinic acid, glucose and fructose were purchased from Sigma-Aldrich (Saint Luis, EUA) and acetic acid, ethanol, dichloromethane and sodium sulfate were purchased from Merck (Darmstadt, Germany).

2.3.2. Minor volatile components

Minor volatile components in the fruit wines were determined by extraction with dichloromethane according to the methods of Oliveira, Faria, Sá, Barros, and Araújo (2006), followed by analysis of the extracts by GC-MS using a Varian 3400 gas chromatograph equipped with a septum-equipped temperature programmable injector (SPI), and an ion-trap mass spectrometer (Varian Saturn II). Samples of 1 uL were injected into a capillary column (Factor Four VF-Wax_{MS} Varian, 60 m \times 0.25 mm i.d., 0.25 μ m film thickness). Helium was used as the carrier gas at 124 kPa (18 psi). The detector was operated in the electron-impact mode (70 eV), and mass spectra were acquired by scanning over the mass/charge (m/z)range of 29-360 with an acquisition rate of 610 ms. The temperature of the injector (SPI) was programmed to run from 20 °C to 250 °C at 180 °C min⁻¹ and was then maintained at 250 °C during the analysis. The oven temperature was held at 60 °C for 5 min, then programmed to run from 60 $^\circ C$ to 220 $^\circ C$ at 3 $^\circ C\,min^{-1}$ and was finally maintained at 250 °C for 25 min.

Volatile compounds were identified using Varian Saturn GC/MS software (Version 5.2) by comparing mass spectra and linear retention indices with those of authentic standard compounds injected under the same conditions. 4-Nonanol was chosen as internal standard and added to each sample and standard to a final

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