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# Good Manufacturing Practices, Hazard Analysis and Critical Control Point plan proposal for distilleries of cachaça

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**ABSTRACT**: *Cachaça* poses many quality (appearance, taste, consumer acceptability) and safety hazards (chemical, metal, and microbiological contaminations during the process). In this work, an effort is made for the quality and safety analysis of *cachaça*, by describing and outlining the potential hazards in every step of the process. This study revealed that the critical control points (CCPs) involved in Good Manufacturing Practices (GMP) to prevent *cachaça* safety hazards are the stages of sugarcane growing, sugarcane harvesting, fermentation, distillation, and aging process. The most significant factors for both CCPs and critical points (CPs) that should be controlled are determined. The implementation of the Hazard Analysis and Critical Control Point (HACCP) system in small distilleries of *cachaça* has been very helpful to provide the required safety for domestic consumers and boost *cachaça* exportations. Therefore, the main objective of the *cachaça* industry is to achieve production consistency.

**Keywords**: quality, microbiological hazards, chemical hazards, sugar cane spirit

## Introduction

*Cachaça* is the Brazilian spirit produced by fermentation of sugarcane juice and subsequent distillation. Attributes of chemical and sensory quality of *cachaça* are totally interconnected to every step of the process, as well as the careful handling of small producers at the main production stages. Good Manufacturing Practices (GMP) establish general principles for all production stages of food and beverages. The presence of chemical hazards poses some safety concerns related to the consumption of *cachaça*. Most hazards originate during the fermentation and distillation steps and are difficult or sometimes impossible to be removed after these steps. In contrast, it is clearly possible to avoid the formation of these substances by applying GMP, Good Hygiene Practices (GHP) and a well-designed Hazard Analysis and Critical Control Point (HACCP) program (Bortoletto and Alcarde, 2015).

In Brazil, controls are submitted to national legislation, under the responsibility of the Ministry of Agriculture, Livestock, and Supply (Table 1). Hazard Analysis and Critical Control Point is a science based on systematic identification of specific hazards and measures for their control to ensure safety of products and processes. Therefore, this procedure is a tool to identify and assess hazards and establish control systems focusing on prevention rather than relying mainly on end-product testing.

Quality is required for the product to meet customer's specifications and is ensured by the application of quality systems. The Codex Alimentarius Commission established the Guidelines for the Application System of HACCP (OPAS, 2006). Every HACCP system can be adapted to changes such as updates in the equipment design, processing procedures, or technological development.

Some principles must be defined and implemented in order to establish the HACCP system in small distilleries. Aiming to comply with the critical limits established by the Brazilian law, some measures have to be associated with each CCP and the critical limits must be controlled in the end product. Methods to check and monitor the process must be established and corrective actions must be taken when critical limits are not met.

Considering these concepts, this study focused on all possiblbe quality and safety hazards, based on HACCP

**Table 1** – Maximum concentration allowed by the Brazilian law for congeners and contaminants in sugarcane spirits and *cachaça* (MAPA, 2005).

| Compound | Legal limit |
|---|---|
| Alcohol content[a] | 38-48 |
| Volatile acidity (acetic acid)[b] | ≤ 150 |
| Aldehydes (acetic aldehyde)[b] | ≤ 30 |
| Esters (ethyl acetate)[b] | ≤ 200 |
| Furfural + HMF[b,c] | ≤ 5 |
| n-propanol[b] | - |
| i-butanol[b] | - |
| i-amylic[b] | - |
| Higher alcohols[b,d] | ≤ 360 |
| Coefficient of congeners[b,e] | 200-650 |
| Contaminants | |
| Methanol[b] | ≤ 20 |
| sec-butanol[b] | ≤ 10 |
| n-butanol[b] | ≤ 3 |
| Copper[f] | ≤ 5 |
| Lead[g] | 200 |
| Arsenic[g] | 100 |
| Ethyl carbamate[g] | ≤ 210 |
| Acrolein | ≤ 5[b] |

[a]% ethanol (v v$^{-1}$) at 20 °C; [b]mg 100 mL$^{-1}$ anhydrous ethanol; [c]5-HMF: 5-hydroxymethylfurfural; [d]sum of isobutyl (2-methyl-1-propanol), isoamyl (2-methyl-1-butanol and 3-methyl-1-butanol), and n-propyl (1-propanol) alcohols; [e]sum of volatile acidity, esters, aldehydes, furfural + 5-HMF, and higher alcohols; [f]mg L$^{-1}$; [g]μg L$^{-1}$.

approach, which may be found during the manufacturing process of *cachaça* in small distilleries, from sugar cane growing to final spirit bottling. These hazards are presented and measures to prevent them are proposed. Also, some possible critical factors and their essential control are approached.

## Methods

A detailed analysis of safety hazards during the whole production process of *cachaça*, including the major steps from sugarcane harvesting to final spirit bottling, was carried out based on HACCP approach. This analysis was basically concentrated on the identification of all possible hazards (physical, chemical, and microbiological), the preventive measures to avoid them, and the detection of CCPs with the respective required control and the relative critical limits.

Similarly, a thorough analysis of quality hazards throughout *cachaça* production process was performed and the critical points (CPs) for the quality of this beverage were identified. The quality and safety results are presented in a table.

## Results and Discussion

The results of HACCP analysis regarding quality and safety hazards during the production process of *cachaça* are presented in Table 2. Preventive and control measures are also proposed in Table 2 for all the potential hazards. The analysis of these hazards and the required controls are based on specialized literature and the practical results of Brazilian distilleries.

### *Cachaça* production in small distilleries

The production process of sugarcane spirits involves the juice extraction from sugar cane using milling tandems. After fermentation, in small distilleries the fermented juice is distilled in pot stills, with the option of maturation in wooden barrels. A flow sheet of the production process of *cachaça* is given in Figure 1.

### GMP and HACCP practices during *cachaça* production

Basic safety procedures to ensure quality of *cachaça* are necessary to comply with the critical limits established for this beverage. These procedures are the foundation to implement HACCP (Table 2).

### Sugar cane growing (CCP and CP)

Stalks of sugar cane (*Saccharum* spp.), a tropical plant originated in New Guinea, Oceania, are the raw material used for the production of sugarcane spirits. The varieties used for the production of spirits are interspecific hybrids primarily developed from the species *Saccharum officinarum*, *Saccharum spontaneum*, and *Saccharum robustum*. Sugarcane stalks are composed of fibers (cellulose, hemicellulose, and lignin – 13 %) and juice (87 %) (Venturini and Nogueira, 2013). The juice contains about 80 % water, 18 % sucrose, and 1 % fructose and glucose. Organic compound non-sugars (proteins, amino acids, lipids and waxes, acids and pigments) and inorganics (mineral salts) altogether represent 1 %. These three types of sugar are converted into ethanol by yeast. In Brazil, sugarcane plantations for the production of *cachaça* cover about 125,000 hectares, resulting in an annual production of approximately 10 million tons of sugar cane (Souza et al., 2013).

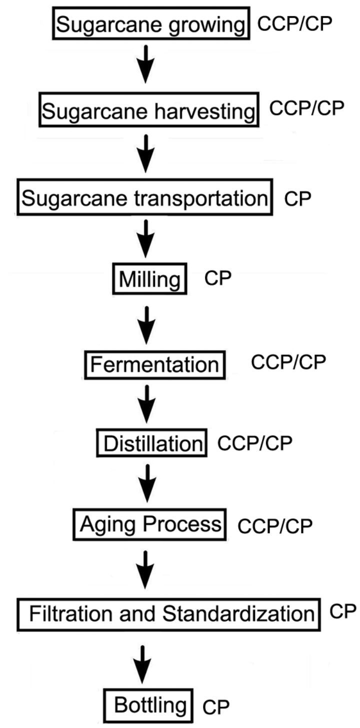

**Figure 1** – Flowchart of the production process of *cachaça*; CCP = critical control point; CP = critical point.

There are several varieties of sugar cane available in Brazil. Choosing the appropriate variety is essencial to obtain raw material with adequate maturation during the ripening season. Sugarcane breeding is carried out by the following institutions: RIDESA – Inter-University Network for Development of the Sugarcane Sector, which produces the RB varieties; CTC – Sugarcaen technology Center, with the CTC varieties and the SP old ones; IAC – Agronomic Institute of Campinas, with the IAC varieties; and CANAVALIS, with the CV varieties (Barbosa, 2012).

Sugarcane, in central-southern Brazil, may be planted throughout the year, but because of the restrictions related to water availability and variety characteristics in maturation and phonological cycle, the main periods of planting are from Sept to early Dec (known as "one-year sugarcane" because this crop will be harvested as early as next season) and from Jan to Mar or Apr (called "one-and-a-half-year sugarcane" because the crop grows for a longer period, around 16 to 18 months) (Barbosa, 2012).

**Table 2** – HACCP analysis during the production process of *cachaça* in small distilleries.

| Step | Hazard type (C, M, P) | Hazard/reason | Preventive measure (GMP) | Monitoring procedure (analysis) | Critical factor/limit/control | Corrective action | Responsible personnel |
|---|---|---|---|---|---|---|---|
| 1. Sugarcane growing (CCP and CP) | | Quality | | | | | |
| CP | M | Fungal, bacterial, and viral diseases of sugar cane | Use of biocides | Use of biocides and pesticides at the correct time | Use at the correct time and according to GAP | Instruction of personnel | Agronomist |
| CP | C | Lack of soil nutrients | Use of fertilizers | Soil analysis for nutrients (specialized laboratory) | Use at the correct time and according to manufacturer specifications | Instruction of personnel | Agronomist |
| | | Safety | | | | | |
| CCP | C | Incorrect use of biocides (dose, type, application time) | GAP | Specific chemical analysis | Proper use according to OPAS (2006) | Instruction of personnel | Agronomist |
| 2. Sugar cane harvesting (CCP and CP) | | Quality | | | | | |
| CP | M | Microbiological contamination or injury | Use of biocides | Analysis of sugarcane juice (reduced sugar concentration) | | Instruction of personnel | Trained personnel |
| CP | M | Bacterial contamination by insects | Use of antimicrobiological agents | Analysis of sugarcane juice (acidity) | | Instruction of personnel | Trained personnel |
| CP | P | Foreign matter from the soil (stones, metal compounds) | Sugar cane harvesting 10 cm above the soil | Visual inspection during harvesting | Regulation of harvest machine and personnel training | Regulation of harvest machine and instruction of personnel | Trained personnel |
| | | Safety | | | | | |
| CCP | C | Formation of furfural, 5-HMF, and PAHs | Avoid sugar cane burning before manual harvesting | Chemical analysis of pure (not aged) *cachaça* using HPLC | Furfural + 5-HMF $\leq$ 5 mg $L^{-1}$ | Abolishment of sugar cane burning before harvesting | Quality control manager |
| CCP | C | Pesticide resid ues | GAP and more care during harvesting | Establishment of the last day of pesticide spraying, use of specific chemical analysis | Proper use according to OPAS (2006) | Delay of harvesting date | Agronomist |
| 3. Sugarcane transportation (CP) | | Quality | | | | | |
| CP | NA | Sugar loss by exudation | Rapid transportation | Chemical analysis | Immediate transportation | Instruction of personnel | Trained personnel |
| CP | M | Increase in microbial contamination and oxidation | Rapid transportation, protection against excessive light and heat | Chemical analysis | Immediate transportation, protection against excessive light and heat | Rejection of contamined sugar cane | Quality control manager |
| CP | P | Foreign matter in sugar cane (residues from the soil, dust) | Adequate harvesting | Visual control | Excessive foreign matter and residues | Sugar cane washing | Trained personnel |
| 4. Milling (CP) | | Quality | | | | | |
| CP | NA | Sugar loss in the bagasse | Mill regulation, bagasse soaking in sequential mills | Chemical analysis of bagasse | Sugar loss $\leq$ 35 % | Bagasse soaking in sequential mills | Quality control manager |
| CP | NA | Must oxidation | Shorter period between harvesting and extraction | Chemical analysis of must | pH 5.4-5.8; acidity 2.0-3.0 g $H_2SO_4$/L | | Quality control manager |
| | | Safety | | | | | |
| CP | C | Grease contamination of mill | Adequate amount of grease | Visual inspection | Abolishment of excessive grease | Mill maintenance | Trained personnel |

Continue...

**Table 2** – Continuation.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| CP | P | Foreign matter in the must from the equipment (metal compounds, screws) | Mill cleaning and use of GMP during juice extraction | Visual inspection | Mill regulation | Mill maintenance | Trained personnel |
| CP | P | Foreign matter in the must from harvesting (sugar cane parts, leaves, soil) | Sieving after juice extraction | Visual inspection | Removal of most harvesting and plant residues | Adoption of an efficient settling system | Trained personnel |
| CP | M | Must contamination due to inadequate cleaning | Adequate CIP (crusher, settling system, sieves, and pipelines) | Visual inspection | Control of mill system cleaning | Daily disinfection of equipment | Trained personnel |
| 5. Fermentation (CCP and CP) | | Quality | | | | | |
| CP | M, C | Presence of chlorine in water to adjust must concentration | Filtration and dechlorination | Analysis of water | NA | Use of filters and demineralization treatment | Quality control manager |
| CCP | M, C | n-butanol and sec-butanol formation | Avoid bacterial contamination and long interval before distillation | NA | NA | Control of distillation process | Quality control manager |
| CCP | M, C | Acetic acid formation by acetic bacteria | Avoid equipment and pump contamination, use of GMP (hygiene and aseptic procedures) | Analysis of *cachaça* using potentiometer and titration | Maintenance of pH 4.0-4.5 and acidity 2.5-5.0 g $H_2SO_4$/L, long interval before distillation | Control of distillation process | Quality control manager |
| CCP | C | Ester and aldehyde excessive formation | Use of proper yeast, avoidance of multiple yeast reuse | NA | NA | Control of distillation process | Quality control manager |
| CCP | C | Higher alcohol formation | Use of proper yeast, control of fermentation temperature, pH, and excessive oxigenation | NA | Maintenance of pH ≥ 4.0; temperature ≤ 32 °C, long interval before distillation | Rejection of batch | Quality control manager |
| | | Safety | | | | | |
| CCP | C | Ethyl carbamate precursor formation (citruline and arginine) | Avoidance of excessive nitrogen fertilization, high temperatures, and nitrogen yeast supplementation, use of proper yeast and GMP | NA | NA | Control of distillation process | Quality control manager |
| CCP | C | Methanol formation | Presence of pectine from bagasse – use of sieve with smaller openings, removal of decanted and floating residues from juice | Visual inspection of residues | Visual inspection | Control of distillation process | Quality control manager |
| 6. Distillation (CCP and CP) | | Quality | | | | | |
| CP | C | Dimethyl sulfide formation, affecting sensory quality | Use of copper equipment for distillation | Sensory analysis of *cachaça* using GC-MS | Sensory acceptance of *cachaça*: DMS ≤ 2.48 ppm | Storage or aging process | Trained personnel |
| CCP | C | High copper content in the final product | Control of pot still cleaning | Chemical analysis | Cu ≤ 5 mg $L^{-1}$ | Rejection of batch | Quality control manager |
| | | Safety | | | | | |

Continue...

TTable 2 – Continuation.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| CCP | C | “Heads” components in excess (acetaldehyde, esters, and methanol) | Control of “heads” cut volume (about 2 % of pot still volume) | Analysis of *cachaça* using GC-FID | Aldehydes ≤ 30 mg 100 $mL^{-1}$ AA; ester ≤ 200 mg 100 $mL^{-1}$ AA; methanol ≤ 20 mg 100 $mL^{-1}$ AA | Redistillation of *cachaça* cutting more the “heads” fraction | Quality control manager |
| CCP | C | “Tails” components in excess (acetic acid and furfural) | Control of “tails” cut volume | Analysis of *cachaça* using GC-FID | Acetic acid ≤ 150 mg 100 $mL^{-1}$ AA; furfural + 5HMF ≤ 5 mg 100 $mL^{-1}$ AA | Redistillation of *cachaça* cutting more the “tails” | Quality control manager |
| CCP | C | Furfural formation | Control of sugar content in the final wine (at the end of fermentation), control of presence of yeast residues | Analysis of *cachaça* using HPLC | Furfural + 5HMF ≤ 5 mg 100 $mL^{-1}$ AA | Redistillation of *cachaça* cutting more the “tails” | Trained personnel |
| CCP | C | Acrolein formation (from pot still or fermentation) | Avoidance of high temperature during distillation, control of “heads” cut volume (about 2 % of pot still volume) | Analysis of *cachaça* using HPLC | Acrolein ≤ 5 mg 100 $mL^{-1}$ AA | Redistillation of *cachaça* | Quality control manager |
| CCP | C | Presence of heavy metals | Use of stainless steel tanks | Analysis of metals using AAS | NA | Rejection of batch | Quality control manager |
| CCP | C | Ethyl carbamate formation | Control of distillation process, temperature, in case of slow distillation, use reflux system in the pot still | Analysis of *cachaça* using GC-MS | EC ≤ 210 µg $L^{-1}$ | Rejection of batch | Quality control manager |
| 7. Aging process (CCP and CP) | | Quality | | | | | |
| CP | M | Microbiological contamination caused by used wooden barrels | Cleaning wooden barrels before use | Microbiological analysis | Absence of yeast, mold, and bacteria | Wooden barrel rewashing | Trained personnel |
| CP | C | Acetic acid excessive formation | Use of well preserved wooden barrels, avoidance of long aging period | Analysis of *cachaça* using GC-FID | Acetic acid ≤ 150 mg 100 $mL^{-1}$ AA | Rejection of batch | Quality control manager |
| CP | C | Reduction of ethanol content | Control of temperature and humidity of warehouse | Ethanol measurement | Minimum of 38 % ABV | Rejection of batch | Trained personnel |
| | | Safety | | | | | |
| CCP | C | Adverse chemical compounds from wooden barrels | Use of proper wood species | Chemical analysis | Certified and proper wooden barrels | Rejection of barrel | Quality control manager |
| CCP | C | HPA and furfural formation due to intensive toasting process | Control of toasting intensity and temperature during cooperage, wash wooden barrel before reusing it | Chemical analysis | Carbonization of wood | Rejection of barrel | Trained personnel |

Continue...

**Table 2** – Continuation.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 8. Filtration and standardization (CP) | | Quality | | | | | |
| CP | C | Turbidity caused by excessive quantity of minerals in water | Use of specific water filters for mineral removal | Chemical analysis | Visual turbidity in standardized *cachaça* | Standardized *cachaça* filtration using a specific filter for mineral removal, cold filtration | Trained personnel |
| | | Safety | | | | | |
| CP | C | Water contamination with heavy metals | Use of specific water filters for heavy metal removal | Chemical analysis using ionic chromatography | Pb ≤ 200 µg $L^{-1}$ and As ≤ 100 µg $L^{-1}$ | Standardized *cachaça* filtration using a specific filter for heavy metal removal | Quality control manager |
| 9. Bottling (CP and CCP) | | Quality | | | | | |
| CP | C | Presence of detergent residues | Control of washing system and water flushing | Avoidance of detergent residues by rinsing several times | No detergent residues | Rinsing several times | Trained personnel |
| | | Safety | | | | | |
| CP | P | Physical contamination (glass, machine parts, insects) | Use of visual control and GMP in bottling area | Visual control | No physical contaminants inside the bottle | Rejection of contamined bottle | Trained personnel |

C = Chemical; M = Microbiological; P = Physical; GMP = Good Manufacturing Practices; CCP = critical control point; CP = critical point; GAP = good agricultural practices; 5-HMF = 5-hydroxymethylfurfural; PAHs = polycyclic aromatic hydrocarbons; NA = not apply; CIP = clean-in-place; HPLC = high performance liquid chromatography; GC-MS = gas-chromatography/mass spectroscopy; DMS = dimethyl sulfide; AA = acetic acid; AAS = atomic absorption spectroscopy; ABV = alcohol by volume; CCPs are in boldface type.

Sugar cane is susceptible to diseases transmitted by viruses (mosaic and chlorotic streak), bacteria (leaf scald, red stripe, and ratoon stunting), and fungi (smut, brown rust, orange rust, red rot, and pineapple rot), which can be inoculated by insect pests (Lima, 2010; Matsuoka and Maccheroni, 2015). The most effective control of these diseasesis the development and use of resistant varieties, along with the use of healthy planting material and biocides (Matsuoka and Maccheroni, 2015).

The major sugarcane insect pests are sugarcane borer (*Diatraea saccharalis*), spittlebug (*Mahanarva fimbriolata*), beetles (*Migdolus fryanus*), and termites. Also, a significant number of phytonematodes can attack this plant. The control of these insect pests can be biological or by using specific insecticides. Sugarcane borer is the most important of all, since it is spread all over the country and causes the greatest losses. This insect pest is biologically controlled using some natural enemies such as *Cotesia flavipes* (Lima, 2010; Macedo et al., 2015).

It is estimated that approximately 1,000 species of weeds are present in sugarcane agroecosystems. The growth of this plant is initially slow, and therefore at early stages, sugarcane is susceptible to weed competition, which can decrease stalk and sugar yield, decrease crop longevity, increase difficulties and costs during harvesting, and decrease industrial quality of raw material. Additionally, weeds can provide home to sugarcane insect pests and diseases. Integrated weed management encompasses preventive control (to avoid entrance and/or spread in the area), cultural control (use of varieties and cultural practices that make the crop more competitive in relation to weeds), mechanical control (hoeing and mechanical cultivators), and chemical control (most used method, because herbicides are efficient and relatively cheap) (Procópio et al., 2015).

Fertilizer use and management in sugar cane includes soil fertility diagnosis, corrective practices (lime, gypsum, and phosphate application), conservationist measures (green and organic fertilization), and mineral fertilization (N, $P_2O_5$, $K_2O$, micronutrients such as zinc, copper, boron, and manganese) (Vitti et al., 2015).

The daily water consumption of the sugarcane crop varies from 2.0 to 7.0 mm. Sugarcane crops require water consumption between 1,500 and 2,500 mm per vegetative cycle (Oliveira et al., 2012). In Brazil, the irrigated sugarcane area is still little expressive, less than 5 % of the cultivated total. This happens mainly because of the high resistance of the crop to water stresss and to geographical location of the sugarcane cultivations, where the rainy season coincides with the vegetative growth, while the maturation phase coincides with the dry period. Nevertheless, the plant responds positively to irrigation in situations where rainfall is not enough of fulfill its water requirement. Sugarcane irrigation brings several benefits, such as increase of stalk productivity and sucrose content, precocity in the harvesntig, and longevity of the sugarcane crop (Oliveira et al., 2012).

**Sugarcane harvesting (CCP and CP)**

Sugarcane diseases can also be inoculated by the tools used to cut and harvest the crop (Lima, 2010). Microbiological contamination of sugarcane juice starts when the plant is cut, because the internal part of the stalk becomes susceptible to the entrance of microorganisms from the soil, air, and sheaths of the plant. Yeast and lactic bacteria are the major microorganisms associated with sugarcane deterioration. These microorganisms may reduce yeast cells viability during fermentation and increase acidity of the spirit, impairing the quality of *cachaça*. The manual harvesting of sugar cane increases the amount of mineral impurities (sand and soil) in the raw material, whereas mechanical harvesting increases the amount of plant parts (leaves and straw) (Lima, 2010).

Precautionary measures are necessary to avoid pesticide residues in the raw material. Furthermore, the waiting period of each chemical used in the crop should be respected to prevent pesticide residues in the industrial process (Macedo et al., 2015).

Chemical compounds used or produced due to sugarcane agricultural management may be present in *cachaça* as contaminants. Furfural and 5- hydroxymethylfurfural (5-HMF) are aldehydes commonly present in *cachaça*, but in excess, they affect quality, conferring a burning taste to the beverage. The limits of furfural and 5-HMF established by Brazilian law refer to non-aged (white) *cachaça* (MAPA, 2005). Polycyclic aromatic hydrocarbons (PAHs), considered genotoxic and carcinogenic, can also be present in *cachaça*, and represent a hazard to public health (IARC, 2010). Although no studies have mentioned the maximum levels of PAHs in spirits in Brazil or other countries, the European Food Safety Authority (EFSA) estimated a maximum daily intake ranging from 6 to 8 ng $kg^{-1}$ of body weight, considering individuals weighing 70 kg (EFSA, 2008). Furfural, 5-HMF, and PAHs result from chemical decomposition of carbohydrates and can originate in different steps of *cachaça* production process.

The Brazilian law limits the concentration of furfural and 5-HMF, because these compounds may be related to sugarcane burning prior to harvesting. Burning makes manual harvesting easier, but causes damage to the environment and is already banned in many Brazilian regions. The heat generated by sugarcane burning causes intense increase in the temperature of the raw material and, consequently, transforms sugars into degradation products, such as furfural, 5-HMF, and some PAHs (Galinaro et al., 2007). Therefore, avoidance of sugarcane crop burning and use of mechanical harvesting should be part of Good Agricultural Practices (GAP).

Furfural and 5-HMF can also be formed by pyrogenation of organic matter in the distillation process in pot stills. To prevent the presence of these undesirable compounds, it is advisable to control the sugar content of the final wine and wait until the end of fermentation (0° Brix) to start distilling it. Some yeast residues in the wine can pass to the pot still and the high temperature of the distillation process can promote the formation of sugar degradation compounds. Nonetheless, most part of furfural and 5-HMF is in the "tails" and can be removed by the early cut of this fraction.

Furfural and 5-HMF also result from Maillard reaction and caramelization during the toasting process of cooperage (Aquino et al., 2006; Bortoletto and Alcarde, 2013; Chatonnet, 1999). Wood compounds (cellulose and hemicelullose) are converted into 5-HMF, 5-methyl-furfural, and furfural. Pleasant aromatic notes, described as "toasted", "caramel", "sweet", and "grilled almonds", are associated with low concentrations of these compounds, and wood toasting plays an important role in their formation (Jaganathan and Dugar, 1999). Therefore, intensive toasting processes promote excessive formation of these compounds and can affect safety of the spirit. GMP procedures should be implemented to obtain barrels that undergo a standardized toasting process, not very intense, which should be washed before new use. Moreover, it is mandatory to monitor 5-HMF and furfural concentrations in *cachaça* during the aging process.

**Sugarcane transportation (CP)**

Sugar cane is a perishable raw material and should be processed up to 24 h after harvesting. Some GMP to prevent early deterioration include rapid transportation to the processing plant, protection against excessive light and heat, prevention of sugar loss by exudation, growth of microorganisms (contamination and oxidation), and presence of foreign matter (e.g. residues of soil, dust) (Souza et al., 2013). Transpiration of harvested cane stalks causes loss of fresh mass because of water loss. Due to the increase of the relative content of fibers, the efficiency of juice extraction decreases during milling.

**Milling (CP)**

In order to preserve the quality aspects during milling, GMP should be applied. Inadequate cleaning of the mill station can be a source of microbiological contamination of the must. To prevent it, a clean-in-place (CIP) program is recommended for this step.

The milling process includes mill regulation and the use of bagasse soaking in sequential mills to avoid sugar loss in this residue (Souza et al., 2013) and physical contaminants in the must (metal compounds of the mill and foreign matter from the harvesting – sugarcane parts, leaves, soil) (Maia and Campelo, 2005). Sequencial mills provide a minimal extraction efficiency of 94 %. A single mill would extract only 60-70 % of sugarcane sugars.

Some safety aspects are linked to contamination of the must with grease from the mill and physical compounds or inadequate cleaning. Grease in the must may promote ethyl carbamate formation (Novaes, 2000). Prevention of must oxidation by ensuring a short period between harvesting and juice extraction may improve fermentation quality.

**Fermentation (CCP and CP)**

Fermentation is considered a CCP due to implications of sugarcane must quality and composition on final characteristics of the spirit, as well as the microbiological community acting during this step (Gomes et al., 2009). Hygienic and aseptic procedures are extremely important to avoid contamination and preserve sensory and safety characteristics of the spirit. For this reason, the sugarcane juice must be sent to fermentation right after its preparation.

The juice extracted during milling may have sugar concentration ranging from 18 % to 22 %. For ideal fermentation, however, the must should contain from 14 % to 16 % of sugars. Thus, the juice dilution with potable water is usually necessary. Water filtration and dechlorination are of paramount importance to preserve yeast performance.

The most common way to conduct fermentation to produce sugarcane spirits is in batches fed with recycled yeast cells obtained through decantation. This process recycles the yeast cells decanted in the wine, which occupy from 17 % to 20 % of the usable volume of the fermentation tank and remain there as inoculum for the next fermentation cycle. It is a way to prevent inoculation with a new amount of yeast every cycle (Souza et al., 2013).

According to ideal practices, the must pH has to be between 4.5 and 5.5 and it does not require nutritional supplementation, since it is a complete medium in terms of all nutrients required by yeast. The alcoholic fermentation must be conducted under controlled temperature (28 °C to 32 °C). Each fermentation cycle normally takes approximately 14 to 24 h. Heating and cooling systems are important to control temperature during fermentation and production of secondary compounds by yeast (Caruso et al., 2008).

Some traditional distilleries prefer to use "natural" yeast (autochthonous non-*Saccharomyces*), because it can have a positive sensorial impact on the typical characteristics of the final product (Gomes et al., 2009). Commonly, non-*Saccharomyces* yeasts present low fermentative performance for ethanol production and most of these microorganisms can produce high levels of acetic acid, acetaldehydes, esters, n-butanol, sec-butanol, and high alcohols. Elevated concentrations of these volatile congeners may affect safety and sensory characteristics of the spirit, without complying with Brazilian identity and quality standards (MAPA, 2005).

Some safety hazards are reduced when the producer uses proper yeast (commercial *Saccharomyces cerevisae*) (Alcarde et al., 2012; Piggott and Conner, 2003) and applies GMP in the fermentation room. The thermal treatment of juice before yeast inoculation plays an important role in physical and microbiological cleaning (Bortoletto et al., 2015). The ideal heat treatment consists of rapidly heating the juice to temperatures ranging from 70 °C to 100 °C, followed by fast cooling in appropriate tanks. This treatment favors the prevalence of the selected yeast inoculated during fermentation cycles.

Volatile congeners are produced during fermentation. Therefore, excessive formation can be controlled by using preventive measures. The content of each volatile congener is measurable only in the final spirit and monitoring procedures are generally not applicable during the fermentation process. Corrective actions are taken during the distillation process in most cases.

The presence of methanol in distilled spirits is undesirable because of its toxicity. This compound is generated during fermentation by the action of yeast pectic enzymes on the galacturonic acid of sugarcane bagasse particles in the juice (Moreira et al., 2012). The elimination of these solid particles can be done by filtering the juice or applying chemical and heat treatments for flocculation, coagulation, and sedimentation of colloids. This way, a clearer juice with smaller contaminant microbial counts is obtained, which is more suitable for fermentation (Schwan et al., 2001). Methanol is concentrated at the "heads" of the distillate and most of it can be removed by controlling distillation or applying double distillation (Bortoletto et al., 2015).

Volatile acidity, measured in terms of acetic acid, is a consequence of bacterial contamination during fermentation. Acetic acid bacteria ferment the wine and increase its acidity (Bortoletto et al., 2015) and lactic bacteria comprise approximately 76 % of the microbiological contaminants frequently found in the production process of sugarcane spirits. Acidity is one of the main causes of sensorial rejection of *cachaça* by consumers (Odello et al., 2009).

The prevention of acetic acid formation is mainly linked to avoidance of acetic bacteria contamination during and after the fermentation step. A CIP system should be applied to equipment and pumps before and after use. Moreover, controlling the "tails" cut during distillation is essential to reduce acidity formed in the wine, and double distillation can be a great alternative to remove excessive acetic acid from the spirit.

N-butanol and sec-butanol are contaminants produced by bacterial action after fermentation, mainly when it requires a long time to start distillation and GMP are not applied. It is impossible to remove these compounds by cutting fractions of distillation. To prevent excessive formation of these compounds, it is mandatory to reduce the period between the fermentation end and the distillation beginning to the minimum possible.

Esters and aldehydes are important components of sensory characteristics in spirits and are related to viscosity and aromatic attributes. Esters are produced by yeast during fermentation, as well as during the aging process because of esterification of fatty acids with ethanol. During the fermentation process, they originate from the esterification of ethanol with acetic acid and the amount produced depends on the relative abundance of the corresponding alcohols and acyl-coA radicals involved in yeast metabolism. Ethyl acetate, the major component of this group, is responsible for the tasty flavor of aged spirits (Litchev, 1989). Aldehydes containing up to eight carbon atoms have untasty aroma and those containing more than ten carbon atoms confer distasteful taste and aroma to beverages. However, the excess of these compounds affects the aromatic balance of *cachaça* and is rejected by consumers. Preventive measures include avoid using autochthonous non-*Saccharomyces* or wild yeast and multiple yeast recycling. The Brazilian law limits the concentration of aldehydes in *cachaça* and it is possible to conduct corrective actions during distillation by controlling the "heads" cut (Alcarde et al., 2014).

Higher alcohols produced by yeast are n-propyl, isobutyl, and isoamyl. Their presence is important for the aromatic characterization of *cachaça*, but when in excess, they cause negative effects. The main strategies for controlling the production of these alcohols are: keeping the ideal temperature during fermentation (28 °C to 32 °C), using proper yeast, controlling the must pH ($\geq$ 4.0), avoiding intense oxygenation in the fermentation tank, and reducing the period between the fermentation end and the distillation beginning. When excessive higher alcohols are formed in spirits, it is impossible to remove them, and the rejection of the batch is strongly recommended.

The most important safety aspect related to fermentation is to avoid the formation of ethyl carbamate precursors. Ethyl carbamate, or urethane, is considered the main contaminant of spirits, since it is a potential carcinogenic compound (EFSA, 2007). The Brazilian law establishes the upper limit of 210 μg $L^{-1}$ ethyl carbamate in *cachaça* (MAPA, 2005). Nevertheless, relatively high concentrations of this compound are generally found in Brazilian sugarcane spirits. In a study, among 268 commercial brands of *cachaça* analyzed, 39 % did not comply with the law limit for ethyl carbamate (Bortoletto and Alcarde, 2015).

During fermentation, the CCP is intended to avert the formation of ethyl carbamate precursors. The presence of ethyl carbamate precursors, such as urea, citrulline, and N-carbamyl phosphates (Riffkin et al., 1989), is related to yeast strains and byproducts of their metabolism (Zimmerli and Schlatter, 1991), fermentation parameters (Reche et al., 2007), such as temperature, alcohol concentration, acidity, pH, and distillation system, light and storage period after distillation (Galinaro and Franco, 2011; Lima et al., 2012; Riachi et al., 2014). Preventive methods include avoiding excessive N fertilization in the soil and any kind of N supplementation in the must, controlling fermentation temperature, and using proper yeast (Bortoletto et al., 2015). For major success, it is crucial to apply GMP.

The presence of contaminants, especially ethyl carbamate, higher alcohols, and volatile acidity, is considered a serious sensory flaw in *cachaças*, inasmuch as they were, according to the findings of Bortoletto and Alcarde (2015), the components that contributed more to the percentage of samples that did not comply with the law. These results suggest that the processing plants are not effectively using GMP to ensure quality of the production process aiming to control quality of the final product.

**Distillation (CCP and CP)**

In relation to wash distillation, small and medium producers conduct it in copper pot stills, while larger producers normally perform it in columns. In small distilleries, the spirit is usually obtained by simple distillation and separation of fractions must be controlled. However, some producers remove a small "heads" fraction during simple distillation and this wrong practice can affect quality of the final product. To date, only a few producers perform double distillation. The first distillation is carried out to recover ethanol from wash, whereas the second distillation is performed with the following cuts in the distillate: "heads" (initial 10 % of the distilled volume), "heart" fraction or spirit (60 % of the distilled volume), and "tails" (final 30 % of the distilled volume).

GMP are applied to distillation mainly to remove or reduce some undesirable compounds produced during fermentation. Aldehydes, esters, and methanol are regarded as "heads" components, because they are more concentrated in the first fractions of the distillate. Their excess can be easily eliminated by controlling the "heads" cut volume (between 1 % and 2 % of the pot still volume). Acetic acid and furfural are concentrated in the last fraction of the distillate and can be removed by cutting about 38 % ABV in the collection of potable spirit (as determined by the demisting test). Demisting test is the exact point for "tails" cut, and it occurs when a fine turbidity is noted in the distillate, due to the presence of a substantial content of higher fatty acid esters (Nicol, 2003).

Concentration of dimethyl sulfide (DMS) above 2.84 mg $L^{-1}$ can affect the sensorial quality of *cachaça* (Odello et al., 2009). The use of copper pot still promotes DMS complexation and, consequently, confers better sensory quality to the final spirit (Toledo and Faria, 2004). However, copper contamination in spirits can occur during the distillation process by dissolving the verdigris formed on the inner wall of the still and internal parts, such as deflegmator and coil of the distilling column. Alcohol and acid vapor can dissolve this compound and contaminate the distillate. High copper contents in the spirit are undesirable, because they are potentially harmful to human health. The Brazilian law established that the maximum permitted content of copper in *cachaça* is 5 mg $L^{-1}$ (MAPA, 2005). Moreover, high copper levels in *cachaça* indicate lack of hygiene. It is recommended to keep the still and streamers filled with water during the breaks that occur in the dry season period. Water reduces copper oxidation, formation of verdigris, and spirit contamination. The first distillation of the season must be carried out with a solution of acetic acid (2 %). Acidity promotes the removal of formed verdigris. The use of solution with vinegar or lemon acids could help producers who do not have access to commercial acid products (Souza et al., 2013).

During the aging process, the use of new wood barrels may be considered to control copper contamination. This metal frequently impregnates the wood and is absorbed by it, which decreases the content of this contaminat during maturation time (Scalbert et al., 1998).

Acrolein is a carcinogenic compound also known as 2-propenal. Formed during fermentation step, it is derived from glycerol dehydration or produced due to bacterial contamination. It is considered an extremely mutagenic substance for humans and animals and its critical limit in *cachaça* is 5 mg $L^{-1}$ anhydrous alcohol. Controlling "heads" cut during distillation is a great measure to avoid the presence of acrolein in the spirit.

Redistillation is a possible technique to decrease the content of undesirable compounds, especially ethyl carbamate formed in the spirit and pot stills with high reflux rates (equipped with dephlegmator or rectifying system), which decreases it by more than 90 % of the total (Alcarde et al., 2012).

Heavy metals can contaminate *cachaça* during the production process, inasmuch as they are present in equipment and tools, in the water used for filtration and standardization, as well as in contaminated equipment during the bottling process. In food companies, all equipment must be made of stainless steel to ensure safety and avoid metal contaminants (ANVISA, 2003). Water supplied to all the plant must be monitored and analyzed every 6 months. It is mandatory to use specific heavy metal filters. Bottling is the last step in the industry and should be well controlled to avoid recontamination of the final product (ANVISA, 2003).

**Aging process (CCP and CP)**

Maturation in wood barrels improves the sensory quality of distilled spirits, and is a necessary step for all noble distillates. Unfortunately, aging is not a mandatory step for *cachaça.* The Brazilian law establishes that aged *cachaça* should contain at least 50 % of the spirit matured in appropriate wooden barrels (maximum capacity of 700 L) for a period of not less than 1 year. Premium and extra premium *cachaça* are spirits completely aged for 1 year and 3 years, respectively (MAPA, 2005).

Oak is the main wood used for spirits aging worldwide because it actively participates in the beverage flavor due to the extraction of aromatic molecules from the wood (Ramirez-Ramirez, 2002). However, native Brazilian woods can be a viable option for *cachaça* producers, since they are easily found and peculiar compounds from each different type of wood may be transferred to the spirit allowing their characterization. The most common wood species for aging *cachaça* are amendoim (*Pterogyne nitens* Tul.), araruva (*Centrolobium tomentosum* Guillem. ex. Benth.), cabreúva (*Myrocarpus frondosus* Allemão), cerejeira [*Amburana cearensis* (Fr. Allem.) A.C. Smith], grápia [*Apuleia leiocarpa* (Vogel) J.F. Macbr.], ipê roxo [*Tabebuia heptaphylla* (Vell.) Toledo], jequitibá [*Cariniana estrellensis* (Raddi) Kuntze], jequitibá rosa [*Cariniana legalis* (Mart.) Kuntze], oak (*Quercus sessilis* Ehrh. ex Schur.), and pereira (*Platycyamus regnellii* Benth.) (Bortoletto and Alcarde, 2013).

In Brazil, about 25 types of national wood have been used for *cachaça* maturation in traditional regions. The Ministry of Agriculture, Livestock, and Supply does not establish specific woods for aging *cachaça* (MAPA, 2005). Several scientific studies in Brazil have assessed the chemical and sensory quality of *cachaça* aged in barrels made of various national types of wood. These studies contributed to the specification and determination of peculiar compounds of each wood type and their effect on the quality of *cachaça*, improving it and avoiding adverse chemical compounds (Alcarde et al., 2010).

Most *cachaça* producers import oak barrels already used in the aging process of other fermented beverages and spirits. In spite of the high alcohol content present in *cachaça*, some flaws can be associated with contaminated barrels (Mosedale and Puech, 1998). The risk of microbiological contamination can be avoided by thoroughly cleaning the barrels before reuse. Additionally, trained personnel may carry out some microbiological analyses to ensure quality of used barrels. Low counts of yeast, mold, and bacteria are desirable (critical limits in Table 2).

The aging process in wooden barrels also induces a gradual increase in volatile acidity and minor volatile compounds due to ethanol oxidation by acetaldehyde and organic acids extracted from the wood (Reazin, 1981). The porosity of some types of wood, resulting from the natural structure of fibers, may allow higher oxidation and, consequently, an increase in volatile acidity (Bortoletto and Alcarde, 2013). In addition to the influence of wood type, the acidification rate is also connected to the age of the barrel. Old barrels tend to promote greater oxidation than new ones do due to weakening of the structure caused by longer and/or more intense ethanolysis reactions (Singleton, 1995).

Generally, the spirit volume and strength are lost due to the evaporation of water and alcohol through the porous wood of barrels (Mosedale, 1995). The wood undergoes a drying process before the barrel construction; therefore, this treatment allows the spirit absorption by the barrel in the first days of maturation. Over time, the dynamic process returns some spirit molecules to the aging beverage, along with wood extractable compounds. Since alcohol molecules are more volatile, they are absorbed more easily than water molecules are, which makes the alcohol content decrease in the early stages of the aging process and slowly return in time. Also, the alcohol content decreases by evaporation to the environment (Singleton, 1995). Losses of alcohol and spirit volume mean economic losses to producers. In order to reduce evaporation losses in warehouses, producers should provide good ventilation, avoid excessive stack of barrels, keep temperature around 20 °C to 25 °C, and keep humidity between 70 % to 90 % (Mosedale and Puech, 1998).

**Filtration and standardization (CP)**

After the aging process, filtration and standardization are important steps to remove any solid particles from the barrels, reduce the alcohol content, and avoid future turbidity in the spirit. Particles deposited at the bottle bottom or suspended in the liquid are considered physical contaminants of this product. To prevent these flaws and ensure a longer shelf live, it is recommended to use good quality water for standardization. Therefore, water should be filtered using specific mineral and filters or heavy metal as a way to prevent turbidity in the final product after bottling. In addition, detergent residues can promote sensory defects in the spirit, causing rejection by consumers. Washing the bottles several times before filling them with *cachaça*, controlling the washing system, and flushing used water are measures that help to avoid residues.

**Bottling (CP)**

According to the Brazilian law, the bottling system must be automatic and the process must be carried out in a specific and separate room at the processing plant (ANVISA, 1993; MAPA, 2005). To check physical contamination (glass, machine parts, insects), it is advisable to carry out a good visual control and apply GMP to the bottling system and bottling area. In all cases of detection of any physical contaminants inside the bottle, it is mandatory to reject the batch.

## Final Remarks

The implementation of HACCP system to small distilleries of *cachaça* has been very helpful to ensure the required safety for consumers in Brazil and boost *cachaça* exportations. Furthermore, it can give support to the main objective of *cachaça* producers, which is to achieve production consistency.

Inasmuch as spirits are comparatively safer than other fermented beverages or foods due to their high alcohol content, the identification of potential chemical hazards and the implementation of preventive and corrective actions are of paramount importance to obtain high quality products. The Brazilian law establishes critical control limits for some chemical compounds and the effective control helps to minimize the outbreaks of incidents that are hazardous for human health. The present analysis is useful to apply HACCP to small distilleries of *cachaça* that have already been using GMP.

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