

Distillation of Brazilian Sugar Cane Spirits (Cachaças)

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1. Introduction

Cachaça is the sugar cane spirit typical of and exclusively produced in Brazil which has alcohol content between 38 and 48% in volume, at 20°C. It is obtained from the sugar cane fermented wort and has peculiar sensory characteristics. It may also include the addition of up to 6g/L of various sugars, expressed as sucrose (Brasil, 2005).

In the production of *cachaças*, as of other distilled beverages, a distillation process is used to isolate, select and concentrate specific volatile components of the "liquid mixture" by heating it (Boza & Horii, 1988; Léauté, 1990). After the fermentation, the distillation is the most important step for the quality of distilled beverages (Janzantti, 2004; Boza & Horii, 1988).

Distillation also promotes some heat induced chemical reactions such as the synthesis of acrolein through a Maillard reaction (Boza & Horii, 1988 ; Nykänen & Nykänen, 1991) and of heterocyclic aromatic compounds, such as furans (furfural, etc.), pyrazines and pyridines (Janzantti, 2004 ; Léauté, 1990). Besides that, distillation causes the extraction of certain long chain esters retained in the yeast cells at the end of the fermentation step, transferring them to the distillates (Nykänen & Nykänen, 1991).

The *cachaças* "wine" composition is quite complex and contains liquid, solid and gaseous substances. Ethanol is the main liquid component, with 5 to 8% v/v, and water is the substance present in the greatest amount - about 89 to 92% v/v. Other liquid components present in smaller amounts are glycerol, lactic acid and butyric acid; volatile components such as esters, acetic acid, propanoic acid, aldehydes and higher alcohols, among others (Novaes, 1999; Bruno, 2006 ; Nascimento et al., 1998a,b).

At every production of a distilled beverage, each volatile component will be more easily distilled according to three criteria: solubility in alcohol or water, boiling point and alcoholic content variation in the vapor phase during distillation (Janzantti, 2004 ; Léauté, 1990).

According to the components' volatility, it is possible to isolate the volatile (water, ethanol and others) components from the non-volatile ones (suspended solids, minerals, yeast cells, non-fermentable sugars, proteins, etc.) obtaining two fractions, phlegm and vinasses, the residue from the distillation of the "wine". The phlegm, main distillation product, is an

impure hydroalcoholic mixture, and its content depends on the type of equipment used (Mutton & Mutton, 2005).

2. Distillation system and practices

Cachaças can be produced by two very different systems: “continuous” and “by batches”. In the traditional continuous system the distillation column used is continuously fed with the “wine”, while in the “by batches” system, typical of alembics, the whole wine volume to be distilled is transferred to a pan before the distillation starts (Bruno, 2006).

The different distillation systems are frequently used to distinguish the type of *cachaça* and the production method. Therefore, *industrial cachaça* would be the one produced from distillation columns of any size, through a continuous process, and *alembic cachaça*, the one prepared in copper alembics, with a limited volume. Among the practices that do not fit in the description of the last process, we can mention the sugar cane burning and the use of chemical adjuncts in the fermentation (Tonéis & Cia, 2005; Ampaq, 2006).

As the distillation process is modified, samples of the same “wines”, distilled in the same type of equipment, produced distillates with different quality. A slower distillation results in higher yield and in an increase of the aromatic compounds content, such as of aromatic esters. A fast distillation reduces the yield and produces distillates that are more acidic and contain an excess of higher alcohols (Almeida Lima, 2001; Mutton and Mutton, 2005).

Reche et al, (2007), use Principal component analysis (PCA), linear discriminant analysis (LDA) and ethanal, ethyl carbamate, dimethyl sulfide, isobutyl alcohol, n-propanal, copper, ethyl acetate, and phenylmethanal as chemical descriptors, to develop a model that showed 95.1% accuracy in distinguishing between *cachaças* distilled in copper pot stills and *cachaças* distilled in stainless steel columns. First, an exploratory analysis was carried out by PCA using analytical data for 82 samples (55 samples of *cachaças* distilled in copper pot stills and 27 samples distilled in stainless steel columns) to verify group formation and data structure. Afterwards, LDA was used for classification purposes. The training set used in the LDA was composed of the 82 samples used in the PCA. The self-consistency of the LDA model was examined by cross-validation using 33 unknown samples. During cross-validation, one sample at a time (of n samples) is left out, and the prediction ability is tested on the sample omitted. This procedure is repeated n times, resulting in n models, and will give an estimate of the average prediction ability for the n models.

2.1 Distillation by batch or intermittent: Alembics

As seen in Figure 1, the common alembic has only one pan, usually made of copper or stainless steel, in which the “wine” is heated to boiling then distilled. This equipment, also called distillation apparatus, can include columns with various external geometric characteristics and internal rectifiers (use of plates or trays, bubble traps, etc.), various extension tubes and cooling systems (Bruno, 2006; Maia, 2000; Pinto, 1986).

The distillation kettle (or pan) heating process may be direct or by steam. This process should be slow and gradual, since abrupt heating of the “wine” may cause the apparatus to overflow.

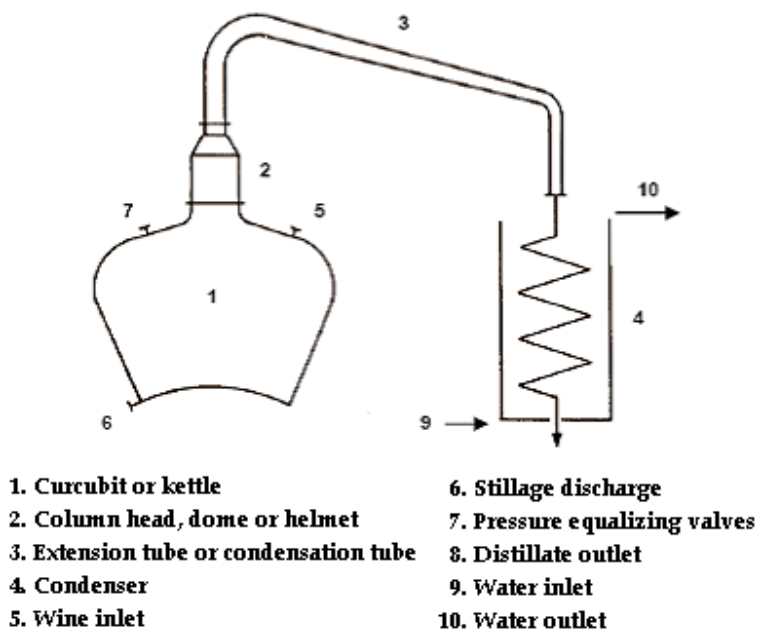


Fig. 1. Common Alembic (Source: Nogueira & Venturuni Filho, 2005)

Besides that, a gradual temperature increase allows the production of vapors which, upon reaching the column head, will partially condense and return to the kettle. The porting of uncondensed vapor reaches the extension tube and partially condenses when becomes in contact with a colder surface. Then it reaches the cooler, where the condensation is completed.

The distillate shows a high alcoholic content (65 - 70% vol) at the beginning of the process and a separation of 5 -10% of the total theoretical volume of spirit by the initial distillation is recommended. This is known as the "head" distillate, rich in aldehydes, ethyl acetate, fatty acids, ethyl caprate and ethyl caprylate (Léauté, 1990; , Mutton & Mutton, 2002; Bruno, 2007), dymethyl sulphide (Nicol; Faria et al., 2003) and other volatile compounds that have greater affinity for ethanol than for water, a factor of greater significance than the individual boiling temperatures (Maia, 2000; Leauté, 1990). The fraction known as "heart distillate", the core distillate, is isolated next and contains a smaller proportion of the "head fraction" components, such as esters, aldehydes, higher alcohols, besides ethyl lactate and the fraction of long chain volatile acids and other undesirable secondary products in concentrations higher than those recommended, formed during the fermentation or inside the alembic itself (Janzanti, 2004 ; Léauté, 1990 ; Mutton & Mutton , 2005). The "heart fraction" represents about 80% of the distillate volume. Since it contains the smallest amount of undesirable substances, this is the best fraction. In practice, usually the phlegm content is controlled around 45 - 50% vol in the receiving box and then the "cut" is made.

Finally, the components with higher boiling points and greater affinity for water are removed. This fraction ("tail") has a high content of phurphural and of other less desirable

components, such as acetic acid, and of the “heavier” fraction of higher alcohols, known as “fusel oil” (Mutton & Mutton, 2005; Nykänen & Nykänen, 1991). The “tail fraction”, also called “weak water”, corresponds to 10% of the distillate total volume, and is collected from an alcohol content of 38% vol up to 10% vol, approximately.

The practices of mixing the “head” and/or “tail” fraction with the new wine (Mutton & Mutton, 2005; Novaes, 1999) are extensively used, either for the recovery of alcohol or to allow the reactions among the “head” fraction compounds (acetaldehyde, ethyl acetate) and the “tail” (acetic and lactic acids) compounds with the wine alcohol, producing aromatic components important for the quality of the *cachaça*. In fact, the richer aroma of the *cachaças* distilled in alembics is related not only to the concentration changes that occur during the distillation processes, but also to the chemical reactions that occur among the components in contact with the hot alembics walls (Faria et al, 2003). These reactions are also favored by the presence of copper and by direct heating (Faria et al., 2003, Léauté, 1990; Nicol, 2003). For such, the distillation should be well conducted, allowing the isolation of compounds that are undesired and harmful to human health (Mutton & Mutton, 2002).

Besides the alembic shown in Figure 1, there are also two or three bodies alembics, which are common alembics adjusted accordingly, in order to save fuel, facilitate and accelerate the distillation process (Maia, 2000; Bruno, 2007).

Figure 2 shows a version of an alembic (2) with two pans. The pans on the right side function as heat exchangers, both to pre-heat the “wine” (up to 60°C) that will be distilled in the second pans at the left, and to pre-cool the emerging vapors condensed on the extension tube, simultaneously.



Fig. 2. Two two-body alembics with direct heating (CQE A and CQE B, Paraty-RJ)

The system may also include one more kettle or pan, making a three-body alembic, as shown in Figure 3. The upper pan works as a heat exchanger, such as in the two-body alembic. The apparatus is fed through the “wine” heater (1). The three bodies are interconnected by pipelines and valves and are at different levels, the “wine” falls by gravity and accumulates in the lowest body, either the exhaustion pan or kettle (2). When the working level is reached (75% of the total volume), the wine starts to accumulate in the distillation pan (3). Once the operative load is completed (75%), the “wine” heater is equally loaded.

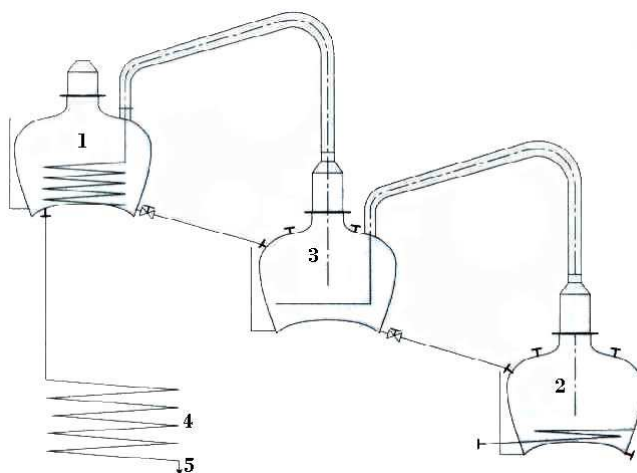


Fig. 3. Three-body alembic (Source: Mutton & Mutton, 2005)

When the valves of the three bodies are closed, the wine in the exhaustion pan is heated by steam or direct heat. The hydroalcoholic vapors containing the most volatile components produced during the progressive heating are, then, injected into the “wine” in the central pan. This enriched and heated wine will produce hydroalcoholic vapors, whose alcohol composition is richer than in those vapors received in the exhaustion kettle (Novaes, 1999). Part of the vapors, richer in water, condenses in the column and returns to the “wine” from where it came, while the fraction richer in alcohol passes through this condensation zone as vapor, reaching the serpentine coil in the “wine” heater, where it condenses. The recently condensed distillate then goes to the cooler serpentine coil (4), and is collected in the reception box (5).

When the alcohol content of the distillate collected in the reception box reaches values between 45 to 48% vol, the heat source and the distillation cycle are interrupted. The vinasses in the third body, which will not contain alcohol anymore, runs out and it will be completed with the distillation kettle “wine”. The kettle is filled with the pre-heater “wine”, which will then be fed with the new “wine”. A new distillation cycle starts with the introduction of steam in the exhaustion kettle (Novaes, 1999 ; Bruno, 2006).

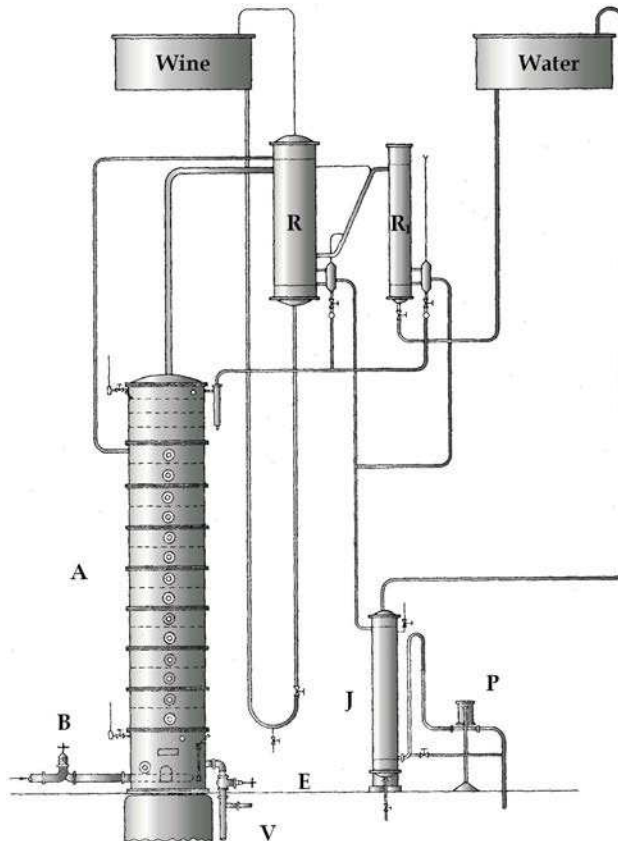
As the distillation progresses, both the uncondensed gases and the future “head” fraction resulting from the “wine” contained in the heater may be removed. Therefore, it will not be necessary to remove them in the next cycle. The “tail” fraction does not have to be removed

either, since the vapors that would generate it are those produced in the exhaustion kettle, which will join the “wine” in the distillation pan (Novaes, 1999).

2.2 Systematic or continuous distillation

Many distillation columns have simply been adapted from those used in the production of ethyl alcohol and produced phlegms with high alcohol content, resulting in products poor in flavor. The distillation apparatus that were made of copper started to be made of stainless steel, at a lower cost. The distillates started to have strange odors, which disappeared inside the vat. However, storage time in the vats was not always enough for that.

In order to eliminate these problems, the distillation columns were redesigned to produce low degree, less rectified phlegm, with enough congeners' content to produce a more favorable *flavor* for marketing purposes (Almeida Lima, 2001). As shown in Figure 4, the column consists of a series of overlapping “plates” or “trays”, which make its core. Two



A- Distillation Column; R - “Wine” heater; R₁ - Auxiliary Condenser; J - Cooler; V - Vinasses; P - Graduate Cylinder; B - Vapor valve, E - Drainage Test.

Fig. 4. Distillation column for *cachaça* (Source: Stupello, 1992)

plates together results in a “segment”. The two trays communicate through a syphon. According to Figure 5, the upper part of this syphon is prominent and conditions the formation of a layer of liquid over the upper tray.

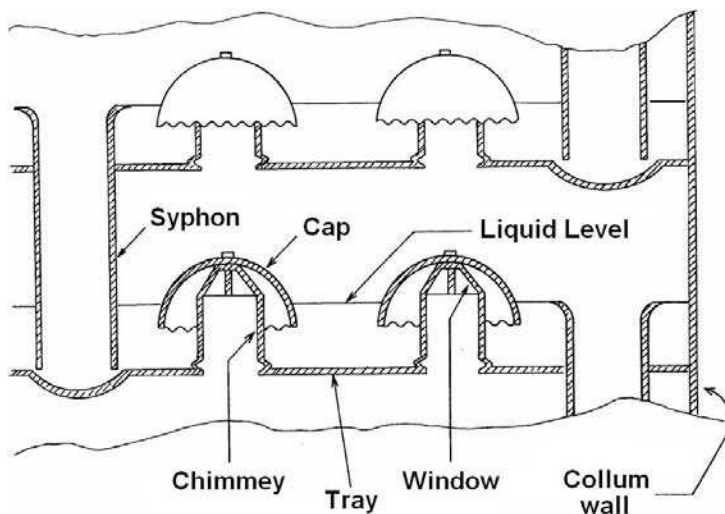


Fig. 5. Scheme of two trays stacked in a distillation column (Source: Novaes, 1993)

Besides the syphon, the tray has a certain number of chimneys stacks with side windows or hatches over which the caps or hats are. The edge of these caps stay immersed in the liquid, offering resistance to the steam passage through the syphon.

The “wine” is introduced in the column (15 to 20 plates) through the upper segment and the steam serpentine coil, for heating purposes, is introduced from the lower segment. To start the process, the “wine” is introduced in the column until it reaches the desired level at the lower segment. The system is cooled with water and the steam is introduced in the serpentine coil. The vapor formed in the lower plate reaches the chimneys of the plate immediately above it and is accumulated in their caps.

Once the pressure increases, vapor starts to bubble in the liquid in the plate, heating and enriching the “wine” on this plate. After some time, the “wine” on the plate above also starts to boil and heats the next plate, and then the “wine” in there and so on until the last plate is reached.

As the phlegm is removed, there is a depletion of the alcoholic content on the plates, with a subsequent increase of the upper plate temperature. When the temperature reaches 92°C, the column starts to be fed again with the new “wine”, setting a continuous flow. The vapor formed in this plate will then be condensed.

System stability is maintained by controlling the vapor inlet, the “wine” flow and the vinasses removal. The use of low alcoholic content “wine” can be worked out efficiently by controlling the condensers downgrading and determining the most adequate selective condition for the final condenser, in order to obtain a better quality *cachaça*. Some compact

distillation columns with a “wine” heater installed at the top of the column make it harder to control the selectivity, and cause the “wine” to be carried to the distillate (Novaes, 1993; Bruno, 2006).

2.3 Bidistillation or double distillation

Bidistillation was effectively introduced in Brazil in 1991, by Novaes (1994), with the purpose of suppressing the ethyl carbamate in the *cachaça* produced at a plant in city of Nova Friburgo (Rio de Janeiro state, Brazil).

The double distillation process in *cachaça* is based on the processes used in the production of whisky and cognac, through the distillation in copper alembics (Novaes, 1994). The first step ends when the distillate inside the collecting box reaches 27% vol, similar to the “weak water”. The vinasses is discarded. This “wine” distillation will be done three or four times until the total “weak water” volume obtained is equal to that of the work load of the alembic.

All fractions from these distillates with low alcoholic content are stored in a flask that has at least the volume of the alembic load, and stay there until the next distillation.

At the end of the last “wine” distillation, the alembic will receive the “weak water” to be distilled. The sample should be heated slowly and, initially, the undesirable gases that do not condense will be released through channeling in the cooler and will be lost into the air. Right after that, a distillate with 77% vol will be collected when the cooling water valve is opened, and kept like that until the end of the process.

Three fractions will be obtained from this distillation: “head”, 1.5% of the “weak water” volume distilled (72 to 75% vol); “heart”, collected in another flask until the alcoholic content reached 66-68% vol; and “tail”, produced until its volume in the collecting box was equal to 30% of the initial “weak water” load.

“Head” and “tail” fractions will be mixed and the volume divided in three parts. Each part will be mixed with a new “wine” load, and so on. The “heart” fraction will be stored in flask according to the type of beverage desired. It can be aged, left to “rest” in adequate vats for a certain period of time, or, simply be consumed *in natura*.

Ethyl carbamate is not too volatile in alcoholic solutions, due to its affinity with water and alcohol. Since it is formed by the distillation process, when a new distillation is done, the product obtained will be poor in ethyl carbamate. Most of the ethyl carbamate will remain in the residue from the first distillation (Riffkin et al., 1989; Novaes, 1994). Similarly, the amount of compounds responsible for carrying copper from inside the serpentine coils (organic acids) is very small, which also results in the production of *cachaças* with low levels of copper and of volatile acidity (Bizelli et al., 2000).

3. Reflux, reflux rate, column geometry and the effect over the *cachaça* composition

Distillation efficiency is controlled by the appropriate column design. The column design is a determining factor for the reflux adjustment, which consists on successive recondensation and revaporization occurring throughout the alembic column. Each time the vapors

condense, a liquid with higher alcoholic content is generated and this liquid is capable of producing new vapors, even richer in ethanol.

At sea level, water boils at 100°C and ethanol at 78.5°C. The boiling temperature for a mixture of these liquids will be closer to 100°C as the ethanol concentration decreases and closer to 78.5°C as the ethanol content increases. A “wine” containing 8% v/v of ethanol starts to boil at a temperature around 94°C and, since ethanol is more volatile than water, the vapors formed will be richer in ethanol (45% v/v) than the mixture that produced them, according to the ethanol-water phase diagram shown in Figure 6. Once the vapors return to liquid state, the ethanol rate will be the same (45% v/v), but the temperature required to vaporize it again will decrease to about 83.5°C.

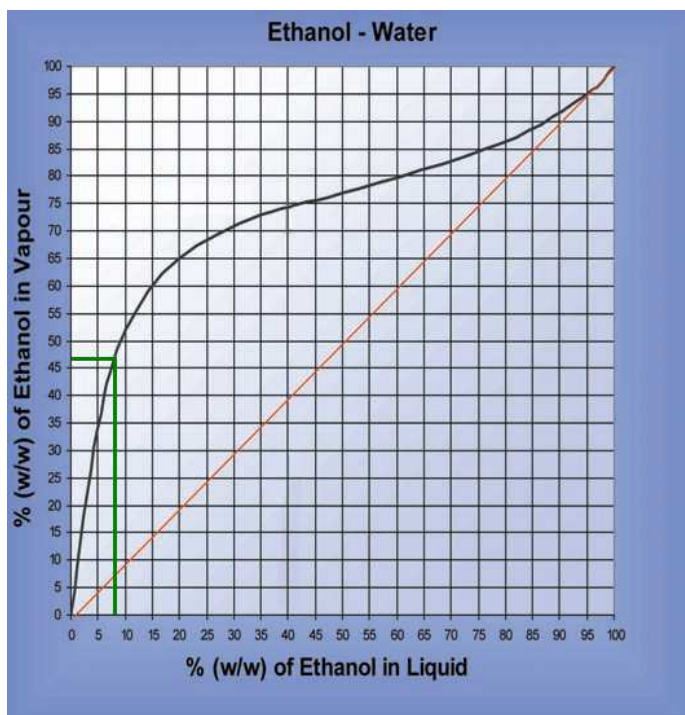


Fig. 6. Phase diagram for the ethanol-water system (Source: Evaporation & Life Science, 2002)

This phenomenon allows the vapors condensed in the column to return to gas phase, even if they stay at a position in the column in which the temperature is permanently lower than that of the liquid in the pan. This is the useful reflux. If the vapors condensed in the column return to the pan, the reflux would have been useless: it will only delay the distillation (Maia, 2000).

The efficiency of the still is a function of its design and of the operation of the column as well as of the reflux apparatus associated with the still head (Maia, 2000). This part of the still divides the condensate so that a portion (D) is taken off as a fresh distillate (P) and

the remainder (L) is returned to the column as a reflux liquid. The ratio L/P is called the reflux ratio. In addition to other factors, the columns give better performance at lower throughputs (Peters et al., 1974) and higher temperature gradients. Under these conditions, the residence time in the column is longer, which results in an equilibrium improvement, distillations with lower temperatures for the ethanol-water vapor phase, which is richer in ethanol than the liquid phase, and in an increased reflux rate (Maia, 2000; Peters et al., 1974).

In the distillation in alembic, the reflux reflects the combined effect of various factors: a) temperature of vapors, from the moment they reach boiling temperature until they reach the access to the extension tube (temperature gradient); b) column geometry and length; c) number and geometry of column plates (Maia, 2000, Claus & Berglund, 2005; Tham, 2006); d) type of bubble traps (Tham, 2006); e) presence and geometry of cooling system on the top of the column (dephlegmator or hood); f) diameter and geometry of extension tube; g) conduction of process (Maia, 2000).

The efficiency of a distillation column separation includes a set of variables, whose complexity makes the appropriate process control less accessible to most of the average *cachaça* producers. Very high vapor or liquid rates can cause problems such as: liquid entrainment, flooding, formation of vapor cones or excess of foaming, among others (Kalid, 2002; Tham, 2006).

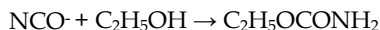
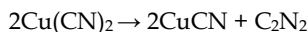
The vapor inlet flow (V_{in}) is generated in the exhaustion section. It is possible to manipulate the reflux rate, equivalent to L, to control the quality of the distillate, because with an increase in reflux, the distillate becomes richer in the lighter products. Likewise, the bottom product composition may become richer in heavier substances, if the rate of heating fluid (water vapor) increases. However, the rate of reflux is a manipulated variable that controls the quality of the distillate (controlled variable), while V_{in} is another manipulated variable associated to the quality of the bottom product. Yet, making measurement in the mixture line is a complex and expensive task. Alternatively, temperature is used to infer the composition. In this case, the controlled variable is the temperature or the temperature difference between two column plates (Kalid, 2002).

4. Influence of distillation systems and processes on the ethyl carbamate content in *cachaças*

Distilled beverages containing cyanogenic glycosides, characteristic of the raw material used, are those in which ethyl carbamate (EC) contents reach the highest values, such as in *brandies* of stone fruits (peach, plum, cherry, etc.) (Schehl, 2005), and *tiquira* (Cagnon et al., 2002), a manioc spirit. The control of the distillation processes for these beverages is not enough to reduce the EC contents to acceptable levels, due to the large cyanide concentration formed. Cyanide is the immediate precursor of EC and this large cyanide concentration cannot be suppressed in the distillate, avoiding the precursors less volatile than cyanides from transferring to the distillates (Bruno, 2006; Lachenmeier et al., 2005). Although sugar cane is considered a cyanogenic plant, cyanide formation in *cachaças* is not very well defined. This is also true for other nitrogen-based EC precursors. However, the main role of the cyanate formed from the oxidation of cyanide, no matter its source (aminoacids, urea, cyanide or carbamylic compounds), has been suggested since the

beginning of the last decade and supported by recent studies and reports (Bruno, 2006; Aresta et al., 2001, Riffkin et al.,1999).

One chemical pathway proposed for the EC formation involves the oxidation of cyanide (CN⁻) to cyanate (NCO⁻) catalyzed by Cu (II) ions (Aresta et al., 2001; Beattie & Polyblank,1995; Mackenzie et al., 1990) followed by reaction with ethanol (Aresta et al., 2001; Taki et al., 1992) according to the reaction scheme below:



Another possible mechanism involves the formation of isocyanic acid, which is released directly from the thermal decomposition of urea present in the wort. Aresta et al., (2001), have reported other possible mechanisms, most of them involving cyanide, copper and cyanate.

According to Riffkin et al., (1989), who used an experimental copper alembic to produce whisky "low wines", the amount of EC formed in the first two hours after distillation, in the presence of 0.8 mg/L of copper, represents about 20% of the EC's final concentration. Approximately 80% of EC were formed within 48 hours in the fresh distillate. By using an Amberlite IRC ion exchange resin a complete inhibition of the EC formation was also verified in fresh distillates when copper ions were suppressed.

Double distillation has been the most common procedure for removing ECs from distilled spirits. Nevertheless, this procedure generally leads to losses in ethanol yield and aroma. Boscolo, (2001), analyzed 84 samples of *cachaças* from various Brazilian regions, with EC levels varying from of 42 to 5689 µg/L and an average value of 904 µg/L. Only 13% presented EC levels below the maximum international limit established (150 µg/L). Those authors reported the occurrence of smaller amounts of EC in Brazilian sugar cane spirits obtained from distillation systems in which the descendent parts (end part of adapter and serpentine coil) are made of stainless steel. However, these systems produce spirits with sensorial defects, mainly due to the presence of sulfur compounds (Faria et al., 2003; Andrade-Sobrinho et al., 2002)., In alembics made entirely of copper, these off-odors usually react more effectively with this metal producing an odorless salt (Andrade-Sobrinho et al., 2002).

A preventive action commonly reported for the reduction of the EC levels in distilled spirits consists in fitting the upper portion of the columns with either a bubble cap tray or a section packed with copper rings (Bujake, 1992) or other copper devices (Andrade-Sobrinho et al., 2002). However, Andrade-Sobrinho et al. (2002) reported the presence of high levels of EC when the distillation system has a small area made of copper in the ascendant parts. They used 126 samples of commercial *cachaças* from several states and reported an average value of 770 µg/L. The average value of the *cachaça* samples produced using distillation columns was of 930 µg/L, whereas those from alembics showed an average of 630 µg/L.

Bruno et al., (2007), studied the influence of the distillation systems and of the distillation process on the EC levels. They analyzed 34 sugar cane spirit samples from 28 main producers (total of 30 different distillation systems) from various regions of the Rio de Janeiro State, Brazil. The first 17 samples consisted of freshly distilled fractions collected in glass flasks from 13 different distillation systems (2 producers have 2 alembics) and four (4) samples of them were recollected afterwards. Samples from alembics were collected from the heart fractions. Seventeen (17) samples were bottled *cachaças* acquired directly at the distilleries. The selection criteria of the 28 producing locations were their different distillation systems, their legal condition, economic importance and the quality of the facilities. These systems were divided in 7 continuous distillation columns (DC); 16 alembics made entirely of copper (CA, pot still); 5 stainless steel alembics with a copper serpentine coil (SSC); 1 alembic made entirely of stainless steel (SSS); and 1 double distillation copper alembic (DDC).

As shown in Table 1, only one out of the seven medium sized continuous distillation columns produced EC levels below the maximum limit of 150 $\mu\text{g/L}$. Ten out of the 16 copper alembics complied with this limit. The results also show that the average of EC levels in the *cachaças* from alembics was lower than the average for *cachaças* from distillation columns (Figure 7).

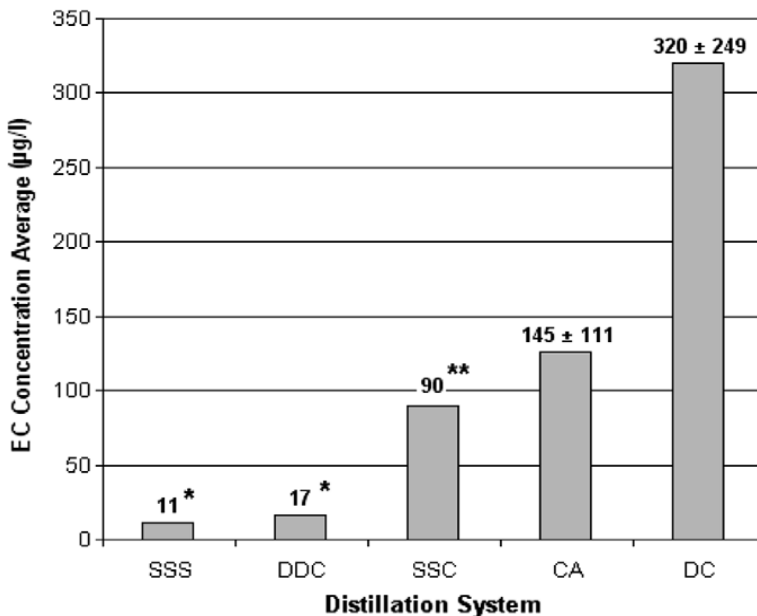


Fig. 7. EC concentration averages for each type of distillation system used for *cachaças* (\pm confidence intervals; $P = 0.05$). *One sample; **non parametric distribution (Source: Bruno et al., 2007)

The results are partially in agreement with those reported by Andrade-Sobrinho et al., (2002), for *cachaças* from various states in Brazil. However, the average for *cachaças* from Rio de Janeiro was 5 to 6 times lower than that described by Boscolo, (2001) and Andrade-

Producer/ Sample	Distillation System/ heating system	EC ± SD (µg/L)	Confidence limits (± µ)	Apparatus coupled (alembics)	Dephlegmator	
EDF	D	CA/DF	<LOQ	-	*	
PTJ	D	CA/DF	45 ± 3	8	*	
PLK	C	CA/DF	214 ± 4	9	*	
JLG (1st)	D	CA/DF	73 ± 1	2	Pre-heater	Tubular (Cu)
JLG (2sc)	D	CA/DF	298 ± 4	10	Pre-heater	Tubular (Cu)
CQE (A)	D	CA/DF	142 ± 1	2	Pre-heater	Bowl jacket
CQE (B)	D	CA/DF	156 ± 3	7	Pre-heater	Bowl jacket
CRD (A)	D	CA/DF	95 ± 6	15	Pre-heater	Bowl jacket
CRD (B)	D	CA/DF	195 ± 6	15	Pre-heater	Bowl jacket
PHM	C	CA/SG	89 ± 2	5	Pre-heater	Tubular (Cu)
SLS	C	CA/SG	50 ± 1	2	Pre-heater	Tubular (Cu)
BVS	C	CA/SG	<LOQ	-	Pre-heater	Tubular (Cu)
SRV (1st)	C	CA/SG	<LOQ	-	Pre-heater	Tubular (Cu)
BAV	C	CA/SG	30 ± 1	5	Pre-heater	Tubular (Cu)
RCL	C	CA/SG	27 ± 2	2	Pre-heater	Tubular (Cu)
CHV	C	CA/SG	599 ± 4	10	Pre-heater + WSA	Bowl jacket
MAG	C	CA/SG	232 ± 5	12	Pre-heater + WSA	*
SBH	D	SSC/SG	79 ± 2	5	*	Tubular (SS)
PRI	D	SSC/SG	35 ± 1	2	*	Tubular (SS)
CTS	C	SSC/SG	17 ± 2	5	*	Tubular (SS)
DNC	C	SSC/SG	215 ± 1	2	*	Tubular (SS)
CPT	C	SSC/SG	106 ± 3	7	*	Tubular (SS)
PCA(1st)	D	DC/SG	714 ± 7	17	*	*
PCA(2sc)	D	DC/SG	456 ± 5	12	*	*
VMC(1st)	D	DC/SG	262 ± 7	5	*	*
VMC(2sc)	D	DC/SG	155 ± 2	5	*	*
BTB(1st)	D	DC/SG	40 ± 3	7	*	*
BTB(2sc)	D	DC/SG	61 ± 2	5	*	*
CAC	C	DC/SG	323 ± 4	9	*	*
SFC	C	DC/SG	252 ± 2	7	*	*
VRI	C	DC/SG	216 ± 6	15	*	*
CAM	C	DC/SG	607 ± 2	5	*	*
SRR	D	SSS	11 ± 2	5	*	*
NFB	C	DDC	17 ± 3	8	*	*

CA: copper alembic; SSC: alembic with stainless steel pot and copper serpentine; DC: continuous distillation column; SSS: alembic entirely in stainless steel; DDC: double distillation in a big pot still used for distillation of whisky; C: commercial cachaças; D: distillates; SG: steam generator; DF: direct fire; WSA = wine stripping alembic; *: absent; (1st), (2nd) - first and second sampling; A, B - alembics A and B of the same producer; SD: standard deviation.

Table 1. Main characteristics and EC levels of the distillation systems evaluated (Source: Bruno et al., 2007)

Sobrinho et al., (2002). About 45% of the products and distilled fractions exceeded the maximum allowed limit of 150 µg/L, with an average of 160 ± 68 µg/L.

During the evaluation of the EC concentrations of 25 brands of pot still *cachaças* produced in the Paraíba State, Brazil, Nóbrega et al. (2009) reported an EC concentration range and average value of 55–700 and 221 µg/L, respectively. EC levels in 70% of the brands exceeded

the international limit for spirits (150 µg/L). The average EC levels found in pot still *cachaças* from Paraíba State are considerably lower than the mean values reported previously for 34 samples of pot still *cachaças* from different parts of Brazil (630 µg/L, Andrade-Sobrinho et al., 2002) and for several commercial *cachaças* produced in Minas Gerais State (1206 µg/L, Baffa Júnior et al., 2007; 893 µg/L, Labanca et al., 2004). Although not clearly stated, the samples investigated by Baffa Júnior et al. (2007) and Labanca et al. (2004) are likely to be pot still types, because Minas Gerais State, in Southeastern Brazil, is the national leader in pot still production of *cachaça*.

However, the average level reported here are higher than the values reported for 13 commercial samples of pot still *cachaças* produced in Rio de Janeiro State (123 µg/L, Bruno et al., 2007).

At first, the lower EC levels found in pot still alembics could be explained by the reactions of cyanide with copper in the ascending parts, which produce non-volatile complexes (Andrade-Sobrinho et al., 2002; Aresta et al., 2001; Boscolo, 2001). However, 2 alembics, without any copper-made part or device inside the columns, produced low EC levels (< 10 and 45 µg/L). These alembics are characterized by a distillation process with low flow rates and temperatures, and suggest that they are favored by their appropriate design (Bruno et al., 2007).

For pot still systems, as the alcoholic content decreases, the temperature of the wine increases. However, it is possible to keep the vapor temperature lower than 80°C in the dephlegmator by increasing the water flow rate inside it during the distillation process. Figures 8.A and 8.B show that it was possible to drastically reduce the EC levels in the freshly distillate fractions from a copper alembic labeled as JLG through this control method. On the other hand, the other 5 alembics that produced lower EC levels (< LQ to 50 µg/L), with the same copper tubular dephlegmator inside the column head of JLG, have two lower sections with stainless steel or copper bubble cap trays (Figure 5 and 9), which leads to high separation efficiencies.

Aiming at reducing the levels of EC in alembic *cachaças*, today, many distillation systems in Brazil are already made with those two lower sections. Therefore, alembic SRV stands out as the one with greater chance of rectification, with three plates with four bubble traps in each plate, made of stainless steel, and a reasonable reflux control system in the dephlegmator. In this system, EC formation was not detected either (< LQ), even when two samples of different batches were analyzed (see Table), which indicated the absence of nitrogenated precursors in more rectifier systems.

In the alembics with a condensing bowl jacket around the still top, it is not possible to properly control the reflux, which, sometimes, can result in distillation temperatures higher than 90°C and accelerate the distillation process. This is the case for the CQE (Figure 2), CRD (Figure 12), and CHV (Figure 13) alembics. The highest EC level among all alembics was found in the CHV unit (595 µg/L), which has in the bottom a wine stripping alembic producing alcohol vapors which will warm it up through the bowl jacket and make the final distillation fraction.

Bruno et al. (2007) also observed that the presence of cloudiness was very common when the temperatures in the column head were above 80-82°C. As a result of entrainment (Tham, 2006; APV,1998), there is an enrichment of less volatile components, such as nitrogenous compounds, into the lower parts of the distillation system (serpentine coil, etc.), which in the

presence of Cu (II) ions and increasing temperatures could eventually promote the decomposition or oxidation reactions responsible for the highest EC levels formed within these systems. These reasons could explain the high EC levels found in the heart and tail samples from the copper alembics (with a pre-heater) CQE and CRD, respectively (Bruno et al., 2007).

As shown in Figure 8.A, the high EC production in the beginning of the first distillation in the JLG (1) could be related to the high initial distillation temperatures (>90°C), which

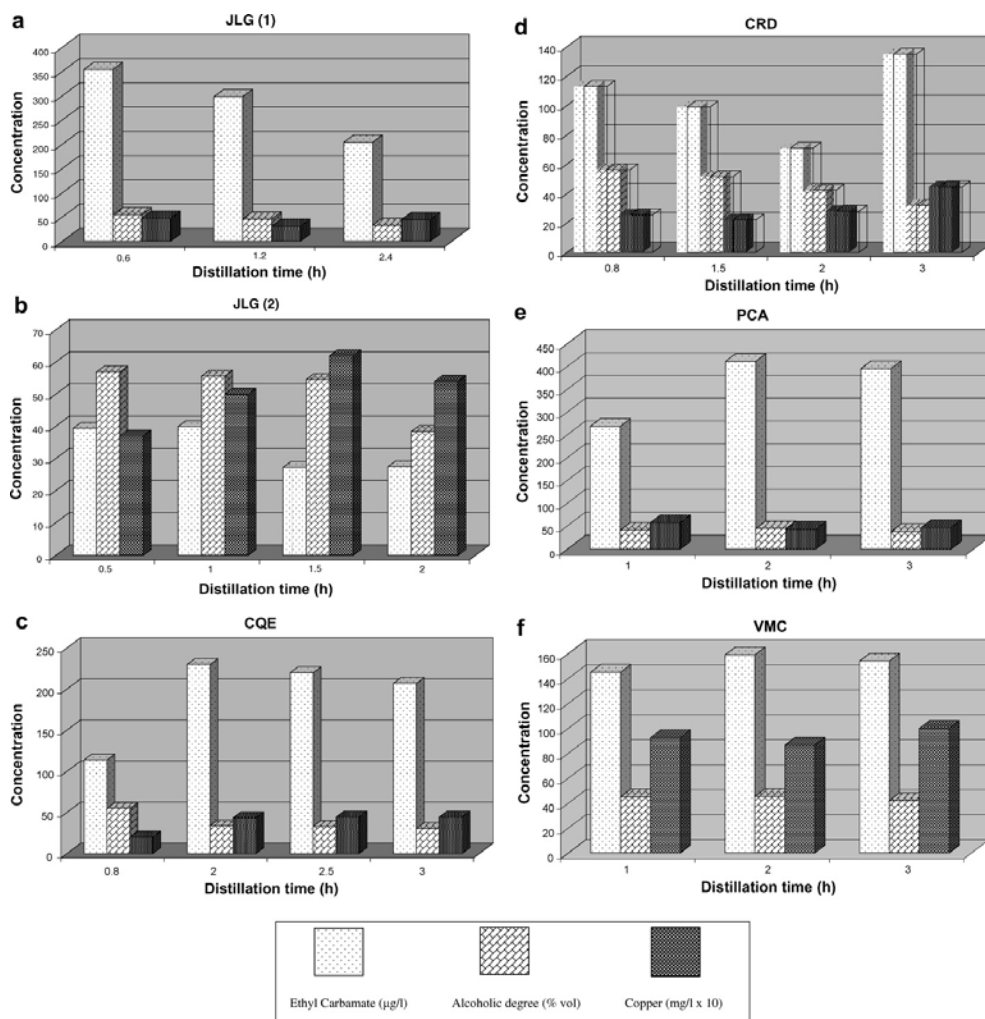


Fig. 8. Ethyl carbamate formation throughout different distillation processes. Cu levels and alcoholic content were measured in the fractions collected at various times during the distillation process. (a) Distillation in JLG with uncontrolled reflux; (b) distillation in JLG with controlled reflux; (c)–(f) distillations in the CQE, CRD, PCA and VMC systems. Each fraction in the histogram was analyzed for ethyl carbamate, ethanol and copper.

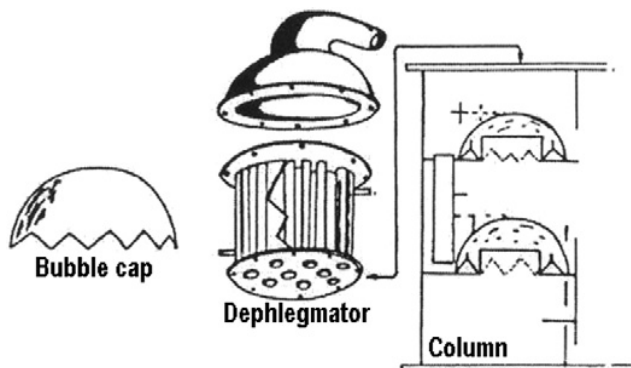


Fig. 9. Scheme of the parts from an alembic column designed to afford an efficient reflux (Source: Unknown).



Fig. 10. Alembic SRV (Valença-RJ).



Fig. 11. Column (at the left) with temperature control by a tubular dephlegmator (alembic JLG ; Bemposta-RJ).



Fig. 12. Details of a cooling system on top of a hood-like column (CRD B, Paraty-RJ).



Fig. 13. Alembic of three bodies (CHV) with a hood-type cooling system (Vassouras/RJ).

promoted the early entrainment previously mentioned and subsequent depletion of the nitrogen precursors. An expressive reduction in the EC levels was observed in the second distillation in the JLG (2) as a result of an adjustment on the reflux, which was obtained by controlling the water flow circulating in the tubular dephlegmator. In this case, the distillation temperatures stayed around 78°C (Figure 8.B).

The lower EC levels seem to come from alembics operating with high reflux ratios, low throughputs and low distillation temperatures (<80 °C). The highest EC levels were measured for those alembics with improper reflux ratios, emphasizing that the reflux ratio plays an important role in the EC formation (Bruno et al., 2007).

In the results obtained by Nóbrega et al (2009), shown in Table 2, with regard to the cooling system, a clear concentration of hot head systems was observed in the most heavily contaminated range (200-700 µg/l). Contrarily, a clear prevalence of dephlegmator and head cooler systems, particularly the former, in the less contaminated range (55-100 µg/l)

Distillery	Brand ^a	EC ^b (µg/l)	Profile of copper pot still distillation ^c			
			Distillation scale ^d	Heating system ^e	Kettle shape ^f	Cooling system ^g (pot still column)
B	02	55	M	SS	O	TD
C	03	60	S	SS	O	TD
D	04	75	M	SS	O	TD
E	05	90	S	DF	O	HC
F	06	100	S	SS	O	HC
M	14	200	S	DF	C	HH
N	15	220	S	DF	C	HH
O	16	225	S	DF	C	HH
G	18	235	S	SS	O	HH
R	24	430	L	SS	B	HH
S	25	700	M	DF	O	HH

^aBrands of white single-distilled cachaças, according to Table 1.

^bEthyl carbamate levels (in increasing order) of cachaça brands.

^cDistillation profile was obtained via data collection during visits to distilleries.

^dDistillation scale, according to total kettle volume of distillery: small (S), 1.000–3.000 l; medium (M), 3.000–9.000 l; large (L), >9.000 l.

^eKettle heating system via internal steam serpentine (SS) or external fire with direct combustion of dried cane bagasse (DF).

^fKettle shape, according to Fig. 1: onion (O), conic (C), and boiler (B).

^gCooling system of pot still column (ascending parts), according to Fig. 2: tubular dephlegmator (TD), head cooler (HC), and hot head (HH).

Table 2. Ethyl carbamate (EC) levels in brands of white single-distilled *cachaças* and their corresponding distillation profile (Source: Nóbrega et al., 2009).

was observed. These observations are in line with those of Bruno et al. (2007), who also found a close connection between low levels of EC in *cachaças* and distillation in alembics using high reflux rates.

The values for the only alembic made entirely of stainless steel agree with those reported by Boscolo, (2001), emphasizing the importance of copper for the EC formation. As expected, the double distilled *cachaça* sample analyzed, produced in a large alembic imported from Scotland, also showed a low EC level. Copper levels in those two systems were 0.01 mg/L and 0.02 mg/L, respectively.

Striking differences in the EC levels measured were also observed for the different continuous distillation columns. Poorly conducted rectification, usually due to the excess of vapor and of wine feeding into the columns (Belincanta et al., 2006; Kalid, 2002) could explain the high EC levels found in most of these systems. In those cases, flooding (in the trays) and entrainment can occur at the same time (Kalid, 2002; Tham, 2006; APV, 1998).

Although there is also a significant variation on the EC levels from different distillation columns, few changes occurred during the distillation processes within these systems (continuous processes) when the temperature did not vary significantly.

4.1 Influence of copper contents on the formation of ethyl carbamate

The EC levels measured as a function of an increase in copper content in the experiment are shown in Figure 1. The initial value of 150 µg/L (in the y axis) was determined from other experiments with various sugar cane freshly distilled samples from other producers, for



Fig. 14. Alembic with stainless steel pan and serpentine coil (Resende-RJ).



Fig. 15. Distillation Column VMC (Bom Jesus do Itabapoana/RJ)

which an average reduction of 65% was obtained and copper levels were reduced to less than 0.01 mg/L (Bruno et al., 2007). In the previous experiment, using another PCA freshly distillate, a maximum reduction of 67% in the EC levels was observed, when the concentration of copper was reduced to 0.16 mg/L. Therefore, in order to achieve a more complete reduction of EC, the copper concentration in the distillates should be around 0.15 mg/L.

About 0.7- 0.8 mg/L was enough to promote a complete EC formation and higher concentrations of copper did not promote any additional catalytic effect.

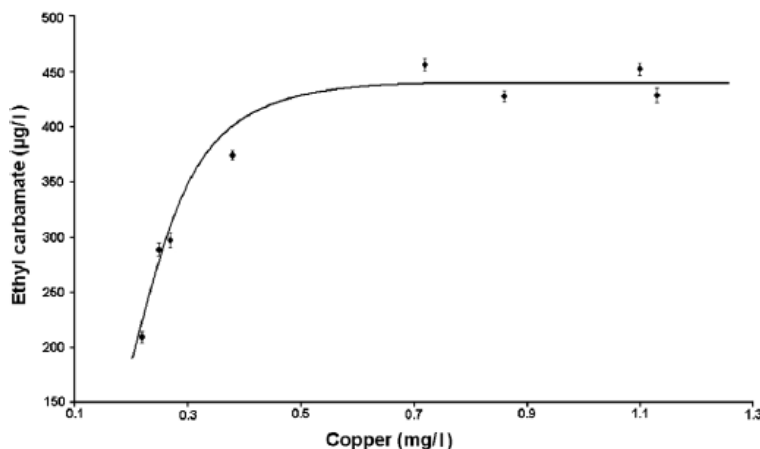


Fig. 16. EC levels as a function of copper concentration in PCA freshly distilled, eluted on Dowex Marathon C resin beds with increasing depths. The error bars show the standard deviations of EC measurements ($n=3$). (Source: Bruno et al., 2007)

5. Conclusion

Some studies on the distillation of cachaças and how it affects the quality of these sugar cane spirits have been published and have contributed to the development of this area. However, there is still a great need for more research on the distillation processes of cachaça in order to continuously improve the quality of these products.

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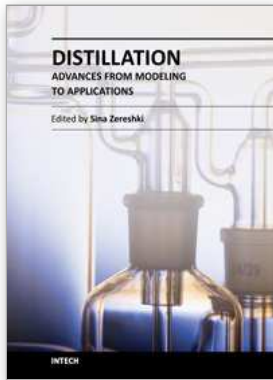
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Distillation modeling and several applications mostly in food processing field are discussed under three sections in the present book. The provided modeling chapters aimed both the thermodynamic mathematical fundamentals and the simulation of distillation process. The practical experiences and case studies involve mainly the food and beverage industry and odor and aroma extraction. This book could certainly give the interested researchers in distillation field a useful insight.

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