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Quality of Distilled Beverages Marketed in Maputo City

António Elísio José[1], Ernesto Boavida Machango[2], Rodrigues Rosário Tranaquela[3]

[1, 2, 3]Higher Polytechnic Instituteof Gaza, Lionde. Gaza Province, Mozambique

Abstract: ***The production of distillate can be summarized in stages such as raw material preparation, transportation, storage, milling, fermentation and distillation. It can, still, suffer aging in wooden containers, bottled and marketed. The present study had as objective the physicochemical evaluation of distilled drinks commercialized in the city of Maputo, quantifying the volatile acidity, by the difference method; alcoholic content, using the method of relative density at 20ºC; methanol, through the oxidation of the methanol in the sample; fixed acidity, by volumetry; total acidity, by the titration method and dry extract by gravimetry. Three brands were analyzed, from local industries in Maputo, consisting of Gin, Liquor, and Whisky. As a result, it was observed a variation of alcoholic degree from 0.01 to 1.10 v/v%, total acidity of up to 0.50 mg of tartaric acid/100 ml, fixed acidity ranging from 0.10 to 1.26 mg of tartaric acid/100 ml, and all samples were free of methanol and volatile acidity. This study concluded that the distilled spirits sold in Maputo city meet the quality standards for alcoholic beverages, and are therefore fit for consumption.***

Keywords: alcoholic beverages, quality control, physicochemical analysis and fraud, Quality of distilled beverages marketed in city of Maputo

1. Introduction

The food analysis is a very important area mainly for the sciences that dedicate themselves in food, because its applicability is so notorious in the various areas of quality control of food, be those during processing, conservation until the consumption of the same. Many times, the term food analysis is simply replaced by other terms such as "food chemistry" and bromatology (EEEP, 2013).

Alcoholic distillate may be considered as a solution of alcohol and water, containing small quantities of secondary components smaller than 1% and which vary according to the composition of each type of alcoholic beverage. The secondary components, or volatile substances or congeneric coefficient, constitute the sum of volatile acidity, aldehydes, esters, higher alcohols expressed by the sum of the same, and furfural; all expressed in mg/100 mL of anhydrous alcohol (IAL, 2008).

The quality and control of brandy are features that are integrated to its importance, therefore require compliance with laboratory analysis, aiming to know the composition both inorganic and organic of it (Volpe, 2013).

An important tool to control the quality of beverages are the physicochemical analyses. Through them, producers monitor physicochemical parameters, maximum and minimum, established. The demand for chemical analysis of beverages has been growing in recent years, mainly by the need to improve the quality standard and industrial control. Only with a strict quality control it is possible to manage the production and monitoring of contaminants from the raw material to the final product (Damasceno *et al*., 2013).

Under these assumptions the present work aimed to evaluate the physicochemical quality of distilled beverages produced and sold in Maputo city.

2. Materials and Methods

Samples of alcoholic beverages consisting of (i) Liquor (ii) Gin and (iii) Whisky, all from local industries in Maputo, were evaluated. The analyses were performed in triplicate at the National Laboratory of Water and Food Hygiene according to the physicochemical methods described by IAL (2008).

2.1. Sample collection

The samples of the beverages, in the state and/or condition in which they are marketed, were randomly collected from 4 producing industries in Maputo province and taken to the National Laboratory of Water and Food Hygiene and stored at room temperature until use.

2.2. Alcoholic strength

The alcohol content was measured using the method of relative density at 20º C, in which 100 ml of the beverage sample was placed in a distillation flask, previously neutralized with 0.1N NaOH in the presence of 3 drops of phenolphthalein as an indicator, the sample was distilled, and the distillate was collected in a 200 ml volumetric flask previously marked to 75 ml. The cooled distillate was transferred to another 100 ml flask and made up to volume with distilled water. A pycnometer previously weighed on an analytical balance was then filled with distilled water and placed with the distillate in a 20ºC thermostatic bath for 30 minutes to stabilize the temperature, after which the pycnometer with the water and then with the sample (the distillate) was weighed and the relative density was obtained according to formula 1.

$$Dr = \frac{m2-m}{m1-m} \quad (1)$$

Onde:
m_1: Mass of pycnometer with distilled water in grams;
m_2: Mass of pycnometer with sample in grams;
m: Mass of the empty pycnometer in grams.

2.2. Dry extract

25 ml of the sample was evaporated to dryness in a porcelain capsule on a 100º C water bath and the remainder was placed in an oven (100º C) for 1 hour, after which the contents were cooled in the desiccator for 30 minutes followed by weighing on a scale ADAM Nimbus®, precision 0.001 g. The dry extract (ES) was calculated using formula 2.

$$ES = (A-b) *20 \quad (2)$$

Where:
A: weight of capsule with dry sample, b: Tare weight of capsule and
ES: Dry stratum.

2.3. Fixed acidity

The dry content, obtained according to the previous procedure, was dissolved in 50 ml of ethyl alcohol (90% in successive fractions) of equal alcoholic strength to that of the sample and transferred to a precipitation beaker containing 200 ml of the water previously boiled and neutralized with an indicator. The mixture was then titrated with 0.1 N NaOH using 2 ml phenolphthalein as an indicator, so the fixed acidity was obtained using formula 3.

$$\textit{Fixed acidity} = \textit{ml NaOH}*30 \quad (3)$$

2.4. Total acidity

For the determination of total acidity, 200 ml of boiled and cooled distilled water was neutralized in a 250ml erlernmeyer flask, where 25 ml of the sample was added, following titration with 0.1 N NaOH and 2 ml phenolphthalein was used as indicator. Formula 4 indicates how the total acidity was obtained

$$\text{Total acidity} = \text{ml NaOH}*30 \quad (4)$$

2.5. Volatile acidity

The volatile acidity (formula 5) was calculated as the difference between the total acidity and the fixed acidity.

$$Av = (At - Af) * 0.80 \quad (5).$$

Where:
At: total acidity, Af: fixed acidity.

2.6. Methanol

Methanol was made by measuring 1 ml of sample into a test tube and adding one drop of $K_2Cr_2O_2$ solution in H_2SO_4 (25:50) and leaving it for 5 minutes at room temperature after which the tubes were placed in an ice bath for 5 minutes. Then 1 drop of absolute ethanol and 3 drops of concentrated chromotropic acid and 5 ml of concentrated H_2SO_4 were added. The presence of methanol was indicated by the appearance of a violet colouration at the test tube interface.

2.7 Statistical analysis

The variance analysis was performed according to the procedures of the SAS statistical software, version 9.0 through the general linear model (GLM), considering the significance level of 5% and the means of the results were compared by the Tukey test.

3. Results and Discussion

As shown in table 1, the alcohol content found in the 3 brands indicated that Liqueur differs significantly from the other drinks, which can be justified by the fact that liqueur is a drink resulting from the mixture of distillate and fruit juice, which possibly contributes to the variation in alcohol content found. Significant difference was also found for dry extract, total acidity and fixed acidity. This difference may be related to the presence of fruit residues in the beverage, which increased the value of dry extract, as well as the acidity of the fruit incorporated in the beverage, which probably influenced the value of the total and fixed acidity of the liqueur.

Table 1: Mean values and standard deviation of physicochemical parameters of distillates and liqueur

Samples	*Alcoholic strength*	*Dry Extract (g)*	*Methanol*	*Total Acidit*	*Fixed Acidity**	*Volatile Acidity*
	(v/v%)		*(P/N)*	*y**		****
Liqueur	17,166±0,01^a	104,110±0,01^a	NE	209,5±0,50^a	270,167±1,26^a	NF
Whisky	41,733±1,10^b	0,482±0,34^b	NE	3,0±0,00^b	3,00±0,12^b	NF
Gin	42,310±0,26^b	0,251±0,00^b	NE	3,0±0,29^b	3,00±0,10^b	NF

mg of tartaric acid/100 ml; ** g of a. Tartaric acid/100 ml; ** g of a. Acetic acid/100 ml, *NF* = Not Found
±Means followed by the same letters in the same column do not have significant differences among them, by Tukey's test at 5% probability.
Source: Authors.

The alcohol content of the liqueur showed the value of 17.16 v/v%, therefore, within the limits imposed by BRASIL (2005) and by Sousa (2015) when indicating that the alcoholic strength of this type of beverages should range from 15 to 54% by volume. Similarly, the samples of brandies, gin and whisky, were also within the limits imposed by INNOQ

(2013), which establishes the minimum of 38 and maximum of 54 % v/v for this type of beverages. These data proved similar to those reported by Pellenz et al. (2017), 37 to 42% ethanol, in a study carried out in MG on the quality of brandies.

The dry extract content ranged from 0.25 to 104.11 g/ml, and the liquor was significantly different ($p<0.05$). The higher value found in liquor sample, probably, is related to its composition, characterized by higher concentration of sugar that, according to Sa (2020) can influence the evaporation in the drying process. Carvalho et al. (2011) obtained, in their study on the evaluation of physicochemical parameters in cachaças, dry extract ranging from 0.192 to 1.718 g/mL, therefore, similar values to those of this work in distillates. Lower values (up to 0.130 g/mL) of dry extract were reported by Pellenz et al. (2017) when studying the quality of cachaça produced in the northern region of mato grosso. Sa et al. (2020) had an average of 27.94g/mL of dry extract, when studying the elaboration of pomegranate and cinnamon liqueur. This average shows lower compared to this research and these authors claim that sucrose, when acting as a solid, dissolves in greater proportion in the syrup, which affects the amount of solids in the dry extract obtained after alcohol evaporation. Santos et al. (2018) observed, during their experiment on the physicochemical characterization of handcrafted produced liqueurs, that when evaporating the entire liquid part of the samples, only a viscous honeydew remained, related to the amount of sugar added to the liqueur, as well as the amount of sugar from the fruit itself added to it, which caused the samples not to dry completely. Carvalho et al. (2011) claim that, the variation of the dry extract in the distillates may be related to the aging time of the distillate, since the newly distilled have lower dry extract contents than the same distillate when aged, the composition of the sample itself, ambient humidity, temperature, and many other factors.
For the total acidity values, the averages ranged from 3 mg of tartaric acid for the distillates to 209.5 mg for the liqueur, and a significant value ($p<0.05$) was also obtained in the liqueur samples. The values for the distillates may be due to the low formation of acetic acid and good manufacturing practices of the beverages, considering that the beverage went through the aging process, which is one of the indispensable factors for deciding the quality of the alcoholic beverage. As for the liqueur, the differentiated value (209.5mg of tartaric acid/100ml) of acidity is possibly due to the high amount (40%) of sugar that the sample presented. Borba et al. (2014) reported, in their study on the determination of alcohol content, acidity and copper in brandies produced in Santo António da Patrulha, total acidity values, 17 to 154 mg/100ml, higher than those found in this research. Penna et al. (2016) ensures that the low acidity content characterizes the brandy as a good quality product.

Regarding the results of fixed acidity, oscillation around 3 to 270.16 mg was observed, without significant differences ($p>0.05$) in the distillate samples. In the Mozambican standard, INNOQ (2013), no limits are established for this parameter. The variation of this parameter tends to increase with the aging period. Similar values to those of this research, with emphasis on brandies, were reported by Borba et al. (2014) when referencing from 1 to 63mg/100mL, when analyzing brandies produced in Santo António da Patrulha. Paz et al. (2007) assume that the fixed acidity is the joint measurement of malic, tartaric, citric, lactic, succinic acids and inorganic acids, For this study, it was perceived that the evolution of these acids was not exorbitant. Esquieri et al. (2009) claim that, fixed acidity has similar behavior to the total acidity, because it tends to increase with the time that the brandy remains in the barrels.

No values were found for volatile acidity in all samples, which possibly indicates optimal condition for consumption, i.e., the analyzed brandies did not suffer microbial contamination during the fermentative process, and all samples were within the limits imposed by INNOQ (2013), which sets a maximum of 150 mg/100 ml of anhydrous alcohol. According to Ribeiro et al. (2016) this parameter is only considered deleterious when values reach values above 150 mg/100 ml. The presence of volatile acidity in a sample is indicative of possible problems related to the manufacturing process. Pinheiro (2010) in his study on the sensory evaluation of the beverages industrial cane spirits and alembic cachaça, had their volatile acidity values between 1.50 to 100, these values are shown above those found in this research. Cardoso (2006) states, in his work on sugar cane brandy, that the volatile acidity levels in brandy are dependent on factors such as the appropriate control of time and temperature during the fermentation process, the type of yeast used, the handling of the must and, especially, the asepsis during the manufacturing process. Considering the arguments of the author previously highlighted, it can be stated that the beverages analyzed in this study were prepared obeying all these factors previously mentioned. Similarly, no evidence of the presence of methanol was found in any sample, thus agreeing with the Mozambican standard that imposes a maximum of 300 mg/100ml of this component in the beverages of the genre. In the work done by Volpe (2013) on artisanal and industrial distillates, the concentration of methanol (0 to 4mg/100 ml) was within the maximum limits of 20 mg/100ml of anhydrous alcohol, established by the Brazilian standard, BRASIL (2005). Corroborating with the present research, Vilela et al. (2007) reported quite low methanol contents (0.001 to 0.029mg/100ml) or non-detected amounts (ND) in all their samples, when studying the physicochemical composition of distillates and their mixtures. Volpe (2013) considers methanol not to be an important component for the sensory characteristics of the beverage, however, its presence in the distillate may cause olfactory aggressiveness, besides being highly harmful to the consumer's health, and for Vilela et al. (2007) this constitutes a good sign, since this alcohol is extremely undesirable and its presence in beverages may be linked to poor filtering of the broth, which enables the presence of berries in the fermentative process, which can be degraded to methanol.

4. Conclusion

It can be concluded that the Gin, Liqueur and Whisky produced in the city of Maputo meet the quality standards defined by the Mozambican legislation, offering safe consumption. The high values of fixed acidity and total acidity recorded in liquor, may be related to the high concentration of sugar present in the samples.

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