Production of a distilled mango drink: volatile compounds and heavy metal

Producción de una bebida destilada de mango: compuestos volátiles y metales pesados

Produção de uma bebida destilada de manga: compostos voláteis e metais pesados

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Abstract

The optimization of processes for the production of fruit distillates is essential to ensure high product quality, comply with current regulations, and promote the development of the agro-industrial sector. The objective of this study was to evaluate the physicochemical properties of a mango distillate (*Mangifera indica* L.), varieties Kent and Alphonso, in order to optimize its production process, the fruits underwent controlled fermentation and subsequent distillation, during which volatile compounds and heavy metals were analyzed. The results showed significant differences between both varieties in terms of the concentration of ethyl acetate and furfural, which influence the sensory profile of the distillate. Methanol concentrations and other harmful compounds remained within regulatory limits. However, the copper concentration exceeded the limit allowed by the Peruvian Technical Standard (5 $mg.L^{-1}$), while zinc levels remained within safe parameters according to the WHO. This study demonstrates that, with adjustments in process control, mango distillate has the potential to be a high sensory quality product suitable for commercialization.

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Resumen

La optimización de los procesos para la producción de destilados de frutas es esencial para garantizar la alta calidad del producto, cumplir con la normativa vigente y promover el desarrollo del sector agroindustrial. El objetivo de este estudio fue evaluar las propiedades fisicoquímicas de un destilado de mango (*Mangifera indica* L.), variedades Kent y Alphonso, con el fin de optimizar su proceso de producción, las frutas fueron sometidas a fermentación controlada y posterior destilación, durante la cual se analizaron compuestos volátiles y metales pesados. Los resultados mostraron diferencias significativas entre ambas variedades en cuanto a la concentración de acetato de etilo y furfural, que influyen en el perfil sensorial del destilado. Las concentraciones de metanol y otros compuestos nocivos se mantuvieron dentro de los límites reglamentarios. Sin embargo, la concentración de cobre superó el límite permitido por la Norma Técnica Peruana (5 $mg.L^{-1}$), mientras que los niveles de zinc se mantuvieron dentro de los parámetros seguros según la OMS. Este estudio demuestra que, con ajustes en el control del proceso, el destilado de mango tiene el potencial de ser un producto de alta calidad sensorial apto para su comercialización.

Palabras clave*:* bebida destilada, mango, propiedades fisicoquímicas.

Resumo

A otimização dos processos para a produção de destilados de frutas é essencial para garantir alta qualidade do produto, cumprir as normas vigentes e promover o desenvolvimento do setor agroindustrial. O objetivo do estudo foi avaliar as propriedades físico-químicas de um destilado de manga (*Mangifera indica* L.), variedades Kent e Alphonso, a fim de otimizar seu processo de produção, as frutas foram submetidas à fermentação controlada e posterior destilação, onde foram analisados compostos voláteis e metais pesados. Os resultados mostraram diferenças significativas entre ambas as variedades em relação à concentração de acetato de etila e furfural, que influenciam o perfil sensorial do destilado. As concentrações de metanol e outros compostos nocivos permaneceram dentro dos limites normativos. No entanto, a concentração de cobre superou o límite permitido pela Norma Técnica Peruana (5 $mg.L^{-1}$), enquanto os níveis de zinco permaneceram dentro dos parâmetros seguros de acordo com a OMS. Este estudo demonstra que, com ajustes no controle do processo, o destilado de manga tem potencial para ser um produto de alta qualidade sensorial e adequado para comercialização.

Palavras-chave: bebida destilada, manga, propriedades fisicoquímicas.

Introduction

Mangifera indica L., varieties Kent and Alphonso occupy a predominant place in the Anacardiaceae family and is a fruit of great commercial importance globally (Sumit *et al*., 2023). The extensive coast of Peru, with its semi-arid climate, is ideal for its cultivation, playing a crucial role in regional economic development (Valverde-Rodriguez *et al*., 2023). However, because it is a seasonal climacteric fruit with a very short shelf life in its fresh form, losses of up to 35 % of the annual harvest have been reported (Mandha *et al*., 2022). Therefore, the production of mango distillates is an effective strategy to reduce losses, which due to its sensory nuances makes this fruit an ideal candidate for the production of distilled beverages (de Oliveira *et al*., 2022).

The production of distillates depends significantly on the efficiency of the fermentation process, because during this stage, microorganisms consume and produce volatile compounds that can alter the sensory profile of the final product (Mandha *et al*., 2022). The first fractions are collected in a range between 66 and 57 °GL and are equivalent to 1 % of the total volume of the distillate, called head, contains volatile compounds such as methanol and ethyl acetate that are undesirable because they are harmful to health and do not contribute to the quality of the distilled beverage (Barchha and Ray, 2023).

Ethanol, the main component of the distilled beverage, can be found from the first fractions of the distillate to the last, but in greater and almost constant proportion in the stage known as body, being responsible for the alcoholic properties of the beverage and, therefore, its commercial value (Marquina *et al*., 2018). But in addition to ethanol, other compounds that contribute to the characteristic aroma and flavour of the distillate may also be present, such as esters and some higher alcohols, provided they are present in adequate proportions (Ibarra-Camacho and León-Duharte 2018).

In the last stage known as the tail are hydrophilic profile acids that function as quality parameters and are regulated by Peruvian legislation due to their impact on the flavour and stability of the distillate (Ibarra-Camacho and León-Duharte, 2018). These contaminants can affect both the safety and quality of the distillate, contributing to the final product not meeting the quality and food safety standards set by regulatory authorities (Hamala and Wierzbowska-Drabik, 2023).

The objective of this research was to determine the physicochemical properties of a distillate of mango (*Mangifera indica* L.), Kent and Alphonso varieties with special attention to the presence of volatile compounds and undesirable contaminants, such as heavy metals, in order to ensure the quality of the final product and to produce a safe and high quality beverage.

Materials and Methods

Raw material

Fruits of *Mangifera indica* L., Kent and Alphonso varieties were purchased at the Arenales market in Ica, Peru (Latitude: 14° 03‘ 40’ S, Longitude: 75° 44‘ 00’ W) and selected based on their maturity which was determined by the percentage of total soluble solids (13 to 16 °Brix), uniform colour, absence of visible infections and mechanical damage. Lalvin DV10 *Sacharomyces cerevisiae* strains and sucrose were obtained from a local trading house.

Preparation of the juice of *Mangifera indica* L.

The mangoes were washed with distilled water and disinfected with a dilute solution of 1 % sodium hypochlorite (commercial chlorine) and then weighed on a digital scale (Import&Export Patrick’s SRL). Subsequently, they were peeled and pulped manually using stainless steel knives, and finally liquefied in a blender (Joseph model, MI, USA). Five replicates per variety were prepared.

The percentage of total soluble solids in both *Mangifera indica* L. juices were adjusted to 22.5 ± 0.5 °Brix with the help of a gum syrup made from commercial sugar. A portable refractometer (model RHB0-80) was used to determine the percentage of total soluble solids. The hydrogen potential (pH) in both *Mangifera indica* L. juices was corrected to 3.3 ± 0.1 pH with tartaric acid in aqueous medium. The pH measurements were performed with a potentiometer (Hanna model HI2210).

Mango juices were inoculated with *Saccharomyces cerevisiae* and fermented at constant temperature (30 ± 1 °C). They were then refrigerated at 4 °C to stop fermentation until distillation in an all-copper distillation apparatus of 5 L capacity.

Determination of volatile compounds

For the determination of volatile compounds, an Agilent Technologies gas chromatograph (model 7890B), equipped with a front autosampler, a Split/Splitless injection port and a DB-WAX capillary column of 30 m length, 0.25 mm internal diameter and 0.25 µm thickness of stationary phase, was used. The initial furnace temperature was set at 50 °C, applying a first heating ramp at 10 $°C.min^{-1}$ to 150 °C, followed by a second ramp of 3 $°C.min^{-1}$ to 280 °C, maintaining this final temperature for 8 minutes, using nitrogen (PA) as carrier gas with a flow rate of 20 $mL.min^{-1}$. The apparatus is equipped with a flame ionisation detector (FID), used for the determination of volatile compounds such as ethyl formate, isoamyl acetate, isopropanol, ethyl acetate, esters, acetaldehyde, furfural, propanol, butanol, isobutanol, 3-methyl-1-butanol/2-methyl-1-butanol, higher alcohols and methanol. The standards used to prepare the calibration curves were of analytical grade and purchased from Aldrich®.

Determination of heavy metals

For the determination of the heavy metals lead, copper, arsenic and zinc, a flame atomic absorption spectrophotometer (Varian brand) was used, using a mixture of acetylene and air as fuel gas.

Statistical analysis

Statistical analysis was performed using Minitab 17 software, evaluating the data using Student's t-test for independent samples, with a confidence level of 95 %.

Results and discussion

Pulp yield

As shown in table 1, there are significant differences (p-value = 0.000) for a 95 % confidence level in the pulp yield of *Mangifera indica* L., per kilogram between the two varieties studied.

Table 1. Pulp yield of *Mangifera indica* L., Kent and Alphonso varieties.

Variety	Yield (% ± s)
Kent	42.68 ± 0.64
Alphonso	46.86 ± 0.46

p_{valor} = 0.000.

As can be seen, the Alphonso variety presented the highest yield (46.86 %). However, in another study, Musyimi *et al*. (2017), reported a juice yield of 72.83 % for the Kent variety, significantly higher than those obtained in this research. This difference may be attributed to the efficiency of the pulp extraction method used, which in their case was mechanical, which could influence the yield obtained.

Fermentation Stage

Alcoholic fermentation is a biological process in which yeast, mainly *Saccharomyces cerevisiae*, transforms sugar into alcohol and carbon dioxide, and during this process several factors can alter ethyl alcohol production by interfering with yeast cellular activity (Côrtes *et al*., 2018). The fermentation temperature for both varieties was kept constant at 30 ± 1 °C, which allowed the juice of *Mangifera indica* L., Kent variety, to reach 10 fermentable ethyl degrees on the tenth day of fermentation, while the Alphonso variety took fifteen days to reach the same level, results similar to those obtained by Côrtes *et al*. (2018), who reported 10 hours of fermentation, for the aqueous extract of Palmer mango pulp at 30 °C, In this regard, Galindo Reyes (2020), indicates that the optimal fermentation conditions are obtained at temperature values between 27 and 30 °C. As can be seen, temperature affects yeast metabolism and consequently fermentation kinetics, which determines the chemical composition and quality of the final product (Musyimi *et al*., 2017).

Physicochemical properties

Total acidity reached 10 $g.L^{-1}$ of tartaric acid for the Kent variety and 12 $g.L^{-1}$ for the Alphonso variety. According to Mandha *et al*. (2022), total acidity values higher than 7.5 $g.L^{-1}$ tartaric acid may indicate fermentation or handling problems.

Regarding volatile acidity, 1.2 $g.L^{-1}$ of acetic acid was obtained for the Kent variety and 1.5 g. L^{-1} for the Alphonso variety, despite the fact that the pH of the mango juice was adjusted to 3.3 ± 0.1 (Galindo Reyes 2020) using tartaric acid, showing that this acid has a lower buffering capacity compared to citric acid, which made it less effective in controlling certain bacteria, as evidenced by acetic acid levels higher than 1.2 $g.L^{-1}$. However, reducing pH below 3.3 with tartaric acid could compromise yeast performance. In this regard, Abud *et al*. (2019), pointed out that this parameter is a key indicator of product quality, since high levels of volatile acidity may indicate microbiological contamination problems or errors in the collection of fractions during distillation, which would negatively affect the sensory profile of the beverage. Similarly, de Oliveira *et al*. (2022), indicated that for fermented fruit juices, levels above 1.2 $g.L^{-1}$ of acetic acid compromise the sensory capacity of the beverage, due to the presence of undesirable microorganisms.

Distillation stage

The distillation process of the two varieties of *Mangifera indica* L. was performed in three stages. The first stage was accomplished when 1 % of the total volume of the distillate corresponding to the head fraction was collected. A second stage, when the distillate was kept at an alcohol content of 40 degrees corresponding to the body fraction and the third stage when the collection of the distillate was stopped when the alcohol concentration started to decrease due to the increase in temperature. According to Muslime *et al*. (2017), this decrease in alcohol concentration is due to the increase of products from other metabolic pathways, such as glycerol and acetic acid.

Determination of volatile compounds in the distillate product

The results obtained for ethyl acetate were 113.6 ± 1.2 mg in the Kent variety and 125.3 ± 0.8 mg per 100 mL of anhydrous alcohol (AA) in the Alphonso variety, showing significant differences (p-value = 0.000) for a confidence level of 95 %. Furthermore, the values obtained are within the range established by the Peruvian Technical Standard (NTP) 211.001 for pisco, which establishes that to ensure the quality and safety of the product, the content must be between 70 and 150 $mg.L^{-1}$ in products such as pisco and other distillates. These results are superior to those obtained by Musyimi *et al*. (2017), who observed that the concentration of ethyl acetate increased from 9.5 to 35.7 mg per 100 mL of AA when the fermentation temperature was slightly increased. While, Abud *et al*., (2019) reported a concentration of 6.9 mg per 100 mL of AA in the body of the distillate made from mango and passion fruit juice, which is considerably lower than the ethyl acetate levels found in the present study.

Ethyl acetate is an ester, known for its contribution to the fruity, sweet and perfumed aroma profile, and is responsible for adding fruity notes to banana and pear, characteristics that are highly valued by consumers (Liu *et al*., 2020). However, in high concentrations, it can generate an unpleasant aroma and taste (de Oliveira *et al*., 2022). In this case, the results showed relatively high concentrations of ethyl acetate, suggesting that, although these mango varieties generate an attractive aroma profile, excessive levels of this compound are unlikely to provide a pleasant aroma and flavour to the beverage. This makes it essential to carefully control the fermentation process to avoid the formation of ethyl acetate, which in high concentrations impairs the sensory profile of the product.

The results obtained for acetaldehyde in *Mangifera indica* L., Kent (15.3 ± 0.9 $mg.L^{-1}$) and Alphonso (13.7 ± 0.9 $mg.L^{-1}$) varieties indicate that there are significant differences (p-value = 0.040) for a confidence level of 95 %. Furthermore, the values obtained are below those established by the Peruvian Technical Standard (NTP) 210.025 for pisco, which establishes that to ensure the quality and safety of the product, the maximum permitted acetaldehyde content is 50 $mg.L^{-1}$. This parameter is also an indicator of ethanol oxidation during the fermentation process and its presence contributes to low quality of the aguardiente (Abud *et al*., 2019).

The results obtained for furfural in *Mangifera indica* L., varieties Kent (3.1 ± 0.1 $mg.L^{-1}$) and Alphonso (5.5 ± 0.1 $mg.L^{-1}$) indicate that there are significant differences (p-value = 0.000) for a confidence level of 95 %. The values obtained for the Kent variety are below those suggested in the Peruvian Technical Standard (NTP) 210.025 for pisco, while for the Alphonso variety they are slightly above the maximum values established by the Peruvian Technical Standard (NTP) 210.025 for pisco, which establishes that the maximum furfural content should not exceed 5 $mg.L^{-1}$. Hatta-Sakoda *et al*. (2024), detected this compound in the last fractions of the distillate, in the alcoholic graduation range between 25 and 9 °GL and called it a tailing compound. This is a potentially toxic compound commonly found in fruit distillates and in small amounts gives the distillate a characteristic aroma (Hatta-Sakoda *et al*., 2024). On the other hand, Abud *et al*. (2019), attribute the increase in furfural concentration with the significant increase in fermentation temperature, which accelerates the sugar degradation processes and attributes a fiery flavour to the distillate. In quantities higher than those reported in NTP 210.025, it can reduce the quality of the spirit, affecting its aroma and flavour, as well as being harmful to the organism (de Oliveira *et al*., 2022).

The results obtained for methanol in *Mangifera indica* L., Kent (44.0 ± 0.9 mg per 100 mL of AA) and Alphonso (61.2 ± 0.9 mg per 100 mL of AA), indicate that there are significant differences (p-value = 0.000) for a confidence level of 95 %. The Peruvian Technical Standard (NTP) 210.025 for pisco establishes that the maximum permitted methanol content is 150 mg per 100 mL of AA, which shows that the results obtained for both varieties are below the standard. Muley *et al*. (2024), indicate that methanol can be formed in the fermentation stage due to the activity of the enzyme pectin methyl esterase present in fruits that catalyses the release of methanol. As pointed out by de Oliveira *et al*. (2022), although it is common to find traces of methanol in fruit distillates, which represents a toxic risk, it is essential to maintain rigorous control of the fermentation process and selection of raw materials. Mansouri *et al*. (2024), observed an increase in methanol concentration from 96 to 123 $mg.L^{-1}$ when the fermentation temperature was increased from 20 to 30 °C. While Galindo Reyes (2020) reported a methanol concentration of 83 $mg.L^{-1}$ when the fermentation temperature was 20 °C. In this regard, de Freitas Pedrosa *et al*. (2023), indicate that raw materials with low pectin content should be considered, as these act as precursors in the methanol formation. Methanol is a highly toxic compound, and its ingestion can have serious effects because it is metabolised in the body into formaldehyde and formic acid, compounds that are extremely harmful and can cause severe damage to health (Mansouri *et al*., 2024). Therefore, the first fraction of the distillate, known as the head, corresponds to the initial 1-2 % of the total volume collected at the start of distillation, which must be carefully separated and removed, as it contains most of the methanol present in the distillate and is strongly influenced by the fermentation temperature.

The results obtained for higher alcohols in the distillate of *Mangifera indica* L., Kent and Alphonso varieties are shown in table 2. The concentration of higher alcohols in the distillate is closely related to the type of yeast used, fermentation time and temperature (Musyimi *et al*., 2017). In addition, they contribute to the improvement of the sensory profile of distilled beverages (de Oliveira *et al*., 2022).

Table 2. Concentration of higher alcohols obtained for *Mangifera indica* L., Kent and Alphonso varieties.

Alcohol	*Mangifera indica* L., var.	
	Kent	Alphonso
Propanol	8.9 ± 0.1[a]	14.9 ± 0.8[b]
Butanol	1.3 ± 0.1[a]	(0.5 ± 0.1[a]
Isobutanol	24.4 ± 1.4[a]	23.0 ± 0.8[a]
3-methyl-1-butanol/2-methyl-1-butanol	87.3 ± 1.4[a]	73.8 ± 0.8[a]
Higher alcohols	121.9 ± 0.9[a]	112.3 ± 0.7[a]

Results expressed in mg per 100 mL of anhydrous alcohol (AA).
Means that do not share a letter are significantly different.

Propanol and butanol are often found in small amounts in alcoholic beverages as impurities, especially when fermentation and distillation conditions are not properly controlled (Arslan *et al*., 2015). The results obtained for propanol in *Mangifera indica* L. distillate, Kent and Alphonso varieties, indicate that there are significant differences (p-value 0.000) at 95 % confidence level between the two varieties. The results presented in table 2 were obtained after a fermentation process at 23 ± 1 °C and subsequent distillation, obtaining results far from the maximum permitted by the Peruvian Technical Standard (NTP) 210.025 with a maximum permissible of 300 mg per 100 mL AA. In addition, the results reported by Musyimi *et al*. (2017), indicate that an increase in fermentation temperature from 20 to 30 °C is associated with an increase in propanol concentration, from 43.9 mg to 52.3 mg per 100 mL of AA. However, the concentrations obtained experimentally at low fermentation temperatures are far from these values, which can be explained by the rigorous temperature control during the fermentation process.

As shown in table 2, for butanol, the results obtained for *Mangifera indica* L., in its two varieties, indicate that there are no significant differences (p-value 0.080) for a confidence level of 95 % between the two varieties. The results were obtained after a controlled fermentation temperature of 23 ± 1 °C and subsequent distillation, obtaining results far from the maximum allowed by the Peruvian Technical Standard (NTP) 210.025 for pisco, which allows a maximum of 100 mg per 100 mL AA, therefore, the exhaustive control of the fermentation temperature contributed, as indicated by Musyimi *et al*. (2017).

As shown in Table 2, for isobutanol, the results obtained for *Mangifera indica* L., in its two varieties, show that there are no significant differences (p-value 0.090) between the isobutanol concentrations in both varieties, with a confidence level of 95 %. Furthermore, these concentrations are well below the maximum limits established by Peruvian Technical Standard (NTP) 210.025, which sets the maximum allowable concentration of isobutanol at 200 mg per 100 mL of AA. Isobutanol can be present in small amounts as an impurity in alcoholic beverages. Galindo Reyes (2020) observed that the concentration of isobutanol increased from 91.4 to 110.7 as the fermentation temperature increased from 20 to 30 °C. The results shown in Table 2 are perfectly related to the sensory characteristics obtained in the distillate in which it was observed that the absence of unpleasant odors negatively influences the flavor and aroma of the distillate (Musyimi *et al*., 2017).

The isoamyl alcohols 3-methyl-1-butanol and 2-methyl-1-butanol are higher alcohols that are considered to be responsible for some of the sensory characteristics of distillates that at high concentrations can negatively affect the organoleptic profile of the product (Song *et al*., 2023). As shown in Table 2, the mixture of these alcohols for *Mangifera indica* L., in its two varieties, shows that there are significant differences (p-value 0.000) between the concentrations in the mixture of 3-methyl-1-butanol and 2-methyl-1-butanol in both varieties, for a confidence level of 95 %. These concentrations are well below the maximum limits established by Peruvian Technical Standard (NTP) 210.025 for pisco, which establishes 400 mg per 100 mL of AA as the maximum concentration allowed for them.

The trace metals detected in the analysis were copper and zinc, as shown in table 3.

Table 3. Concentration of copper and zinc obtained in the distillate of *Mangifera indica* L., Kent and Alphonso varieties.

Metal	*Mangifera indica* L., var.	
	Kent	Alphonso
Copper	8.27 ± 0.10[a]	8.53 ± 0.12[a]
Zinc	0.09 ± 0.01[a]	0.24 ± 0.11[a]

Results expressed in $mg.L^{-1}$
Means that do not share a letter are significantly different.

As shown in Table 3, for copper and zinc, the results obtained for *Mangifera indica* L., in its two varieties show that there are no significant differences between the concentrations of copper (p-value 0.000) or zinc (p-value 0.000) for both varieties for a confidence level of 95 % when the Student's t-test was applied for a significance of 0.05. On the other hand, the Peruvian Technical Standard (NTP) 211.001 for pisco establishes a maximum permitted limit of 5 $mg.L^{-1}$ for copper in alcoholic beverages, and the results for both varieties exceed the limit established by the NTP. As for zinc, although NTP 211.001 does not establish a maximum permitted limit, the World Health Organization (WHO) indicates that the tolerable daily intake of zinc for an adult is approximately 40 $mg.day^{-1}$ (Schoofs *et al*., 2024; Wolf *et al*., 2022), including all dietary sources, and in this case the established limit is not exceeded.

Copper is a material widely used in the manufacture of stills and distillation columns because it is an excellent conductor of heat and has the ability to remove unwanted compounds such as aldehydes and volatile fatty acids during distillation and can have a significant influence on alcoholic beverages (Ratkovich *et al*., 2023). Zinc is an essential mineral for human health and is found in small amounts in many foods. In alcoholic beverages, zinc can be present as a result of contamination or impurities from ingredients, production equipment or packaging (Ibarra-Camacho and León-Duharte, 2018).

It is very important to keep in mind that the practical implications of this research are significant for the agroindustrial industry, since being able to optimize the production of fruit distillates, in this case of *Mangifera indica* L., not only ensures a high sensory quality of the product, but also compliance with international regulations regarding the concentration of volatile compounds and heavy metals. On the other hand, the identification of critical variables, such as the amount of copper and the regulation of compounds such as ethyl acetate, provides a solid basis for improving the efficiency of the production process, which contributes to the safe and competitive commercialization of this type of distilled beverages in the local and international market.

Conclusions

Mango distillate *Mangifera indica* L., in its Kent and Alphonso varieties, proves to be a viable option for the production of alcoholic beverages, especially due to its pulp yields and the physicochemical characteristics obtained after fermentation and distillation. These results indicate that, with adjustments in the process, mango distillate could meet the quality and safety standards for its commercialization, providing a product with attractive sensory potential and added value.

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