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BEBIDA DESTILADA DE FERMENTADO DE UVAS

DISTILLED BEVERAGE FROM FERMENTED GRAPES

BEBIDA DESTILADA DE UVAS FERMENTADAS

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Resumo

Este estudo consiste em avaliar o comportamento de coluna de destilação com 10 pratos perfurados na obtenção de destilado de vinho. O fermentado com 12 GL foi obtido de mistura álcool etílico – água, e alimentou uma coluna com 10 pratos perfurados operando com relação de refluxo 4. Efetuou-se estudo do desempenho da destilação diferencial. Portanto, o objetivo deste estudo foi avaliar o desempenho de uma coluna de destilação de pratos perfurados de planta piloto localizada no Instituto Superior de Engenharia do Porto, em Portugal. Os resultados mostraram que a concentração de destilado, comparada com a destilação diferencial é cerca de três vezes maior, quando a mesma quantidade de destilado é obtida; os dados intermediários são preciosos para subsidiar estudos de destilação de vinho para obtenção de bebidas destiladas.
Palavras-chave: Fermentado; Uva; Destilação; Coluna de pratos; Destilação diferencial.

Abstract

This study evaluates the behavior of a distillation column with 10 perforated plates in obtaining wine distillate. The fermented liquid with 12 GL was obtained from a mixture of ethyl alcohol and water and fed into a column with 10 perforated plates operating at a reflux ratio of 4.. A study of the differential distillation performance was carried out. Therefore, the objective of this study was to evaluate the performance of a perforated plate distillation column in a pilot plant located at the *Instituto Superior de Engenharia do Porto*, in Portugal. The results showed that the distillate concentration, compared to differential distillation, is approximately three times higher when the same quantity of distillate is obtained; the intermediate data are valuable for supporting studies on wine distillation for obtaining distilled beverages.
Keywords: Fermented; Grape; Distillation; Plate column; Differential distillation

Resumen

Este estudio evalúa el comportamiento de una columna de destilación de 10 platos perforados para la obtención de destilado de vino. El líquido fermentado con 12 GL se obtuvo a partir de una mezcla de alcohol etílico y agua, y se alimentó a una columna con 10 platos perforados que operaban a una relación de reflujo de 4. Se realizó un estudio del rendimiento de la destilación diferencial. Por lo tanto, el objetivo de este estudio fue evaluar el rendimiento de una columna de destilación de platos perforados en una planta piloto ubicada en el Instituto Superior de Engenharia do Porto, Portugal. Los resultados mostraron que la concentración de destilado, en comparación con la destilación diferencial, es aproximadamente tres veces mayor cuando se obtiene la misma cantidad de destilado; los datos intermedios son valiosos para respaldar los estudios sobre la destilación de vino para la obtención de bebidas destiladas.
.**Palabras clave**: Fermentado; Uva; Destilación; Columna de platos; Destilación diferencial.

1. Introduction

The production of grape-based distilled spirits represents the convergence of tradition, biochemical transformation, and separation engineering. Renowned beverages such as Cognac, Armagnac, Pisco, and Brandy are distinguished by

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their complex sensory profiles, which emerge from the careful integration of two decisive stages: fermentation and distillation of grape must be carried out. While distillation is responsible for concentrating and refining volatile compounds, the qualitative potential of the final spirit is fundamentally established during fermentation, where ethanol and a complex matrix of aroma-active congeners are formed (LUKIĆ et al., 2020; GARCÍA-LLOBODANIN et al., 2021). Accordingly, fermentation should not be regarded merely as a preparatory step, but rather as the biochemical foundation that governs both distillation efficiency and the sensory identity of the resulting spirit.

Grape must fermentation is a dynamic biochemical process that functions as an aroma-generating bioreactor. Beyond the conversion of sugars into ethanol, yeast metabolism, predominantly driven by Saccharomyces cerevisiae, leads to the formation of esters, higher alcohols, organic acids, aldehydes, and other volatile compounds that collectively define the aromatic framework of the base wine. The concentration and distribution of these congeners are strongly influenced by technological parameters such as fermentation temperature, nutrient availability, particularly yeast assimilable nitrogen, and yeast strain selection, including the use of non-Saccharomyces species in mixed or sequential fermentations (BELDA et al., 2017; BINATI et al., 2020). This biochemical complexity directly determines the behavior of volatile compounds during subsequent separation operations.

Distillation constitutes the critical unit operation that exploits differences in volatility to separate and concentrate ethanol and flavor-active congeners from the fermented must. In batch distillation, the distillate is conventionally divided into heads, hearts, and tails, corresponding to fractions enriched in highly volatile compounds, ethanol and desirable aromas, and less volatile components such as fusel alcohol and fatty acids, respectively (ROSA et al., 2018). The precision of these cut points is highly dependent on the initial composition of the ferment, further highlighting the intrinsic link between fermentation management and distillation performance.

Among batch distillation techniques, simple differential (pot still) distillation and fractionated distillation using perforated-plate columns represent two distinct

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operational philosophies. Differential distillation, governed by Rayleigh’s law and characterized by a single equilibrium stage without reflux, produces a distillate with continuously changing composition and overlapping fractions, requiring skilled operational control (ATHANASIADIS et al., 2022). In contrast, perforated-plate columns operate with multiple equilibrium stages and controlled reflux, enabling rectification through repeated vapor–liquid contact. This configuration enhances separation efficiency, allows greater control over ethanol concentration, and improves selectivity among volatile congeners, albeit at the cost of increased energy demand and operational complexity (TSAKIRIS et al., 2016; GÓRAK; OLUJIĆ, 2019).

The choice of distillation technology has implications that extend beyond product quality. From a technological and economic perspective, separation efficiency directly affects ethanol recovery, energy consumption, and overall process sustainability. Recent studies emphasize the importance of optimizing distillation conditions through process intensification strategies, such as improved column design, reflux optimization, and energy integration, to reduce the environmental footprint of spirit production (ESTEBAN-DECLOUX et al., 2018; MARQUES et al., 2021). Simultaneously, regulatory frameworks and traditional production practices, including the mandatory use of copper pot stills in certain appellations, continue to shape technological choices, balancing innovation with heritage (LÉAUTÉ, 2020; BUTRÓN-BENÍTEZ et al., 2024).

In this context, the present study evaluates the feasibility and performance of distilling fermented grape must using two batch distillation approaches: simple differential distillation and fractionated distillation employing a perforated-plate column. The central hypothesis is that the distillation technique significantly influences separation efficiency, ethanol concentration, and the distribution of aroma-active congeners, thereby affecting both process performance and the hedonic quality of the distillate. Based on these considerations, the experimental work focused on distillation using a perforated-plate column, aiming to assess its potential advantages to produce grape-based distillates with improved operational efficiency and desirable sensory attributes.

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This study relates to the possibilities of producing a grape fermentation distillate with hedonic characteristics. Therefore, the objective of this work is to study the possibility of distilling wine using a perforated plate column or differential distillation equipment. The study is justified by the fact that the technique used to produce the distillate influences the concentration of the product obtained and therefore the efficiency of the separation. However, the study is limited to the binary system: ethanol-water. Based on these considerations, distillation with perforated plates constituted the experimental part carried out. The study consists of an experimental comparison versus a theoretical model.

2. Literature Review

Grape juice fermentation represents one of the oldest biotechnological processes developed by humankind and, at the same time, one of the most complex, as it involves the transformation of grape must into wine through a series of interdependent biochemical reactions mediated by microorganisms. This process is predominantly driven by yeasts of the genus *Saccharomyces*, which are responsible not only for the conversion of fermentable sugars into ethanol and carbon dioxide but also for the formation of a wide range of secondary metabolites. These compounds, collectively known as congeners, include higher alcohols, esters, organic acids, aldehydes, and sulfur-containing compounds, which together define the aromatic and sensory profile of the base wine and, consequently, directly influence the quality of the resulting distillate (VARELA; BORNEMAN, 2017). Therefore, fermentation constitutes a critical stage of sensory modulation, as the nature and concentration of these congeners are governed by controllable technological parameters.

The fermentative dynamics of grape juice are influenced by multiple interrelated factors, including must composition, such as sugars, organic acids, nitrogenous compounds, and minerals, environmental conditions (temperature, pH, and oxygen availability), and the diversity, succession, and interactions of the

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microbial communities involved (MOURET et al., 2021). An integrated understanding of these factors and their interactions is essential for process control and optimization, enabling the consistent and reproducible production of wines with desired chemical and sensory attributes.

From a microbiological perspective, grape juice fermentation is characterized as a complex ecological process marked by the succession of different yeast and bacterial species. The composition and dynamics of these microbial communities directly affect both fermentation efficiency and the chemical and sensory profile of the final product. Among the most critical determinants of ferment quality is yeast strain selection. *Saccharomyces cerevisiae* remains the primary species responsible for alcoholic fermentation due to its high ethanol tolerance, efficient sugar conversion, and ability to dominate the fermentative environment (SIPICZKI, 2022).

Recent studies have demonstrated that different strains of *S. cerevisiae*, as well as non-*Saccharomyces* species such as *Torulaspora delbrueckii* and *Lachancea thermotolerans*, exhibit distinct enological behaviors, producing variable profiles of fruity esters, higher alcohols, and organic acids (BENITO, 2018; BELDA et al., 2017). In this context, co-inoculation or sequential inoculation of non-*Saccharomyces* yeasts with *S. cerevisiae* has been extensively investigated as a strategy to enhance aromatic complexity, modulate acidity, and enrich the sensory profile of the base wine, thereby positively influencing the characteristics of the subsequent distillate (BINATI et al., 2020).

Fermentation temperature is one of the most critical technological parameters, as it directly affects yeast growth kinetics, sugar conversion efficiency, and the synthesis of aroma compounds and secondary metabolites (DEED et al., 2017). Fermentations conducted at lower temperatures, typically between 14 and 18 °C, favor the preservation of volatile aromatic compounds, particularly esters and thiols, whereas higher temperatures, above 25 °C, accelerate microbial metabolism but may result in increased aroma volatilization and enhanced formation of higher alcohols and other heavier compounds (LUKIĆ et al., 2020).

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The choice of fermentation temperature must consider the yeast strain employed, the intended wine style, and the characteristics of the grape variety. Aromatic white wines generally benefit from lower fermentation temperatures, while red wines are often fermented at higher temperatures to promote the extraction of phenolic compounds from grape skins. Deed et al. (2017) demonstrated that, in Sauvignon Blanc wines, fermentations conducted between 12 and 15 °C promoted greater retention of volatile aroma compounds, resulting in more intense and fruity sensory profiles, whereas temperatures between 18 and 22 °C accelerated fermentation but led to increased aroma loss and higher production of higher alcohols at concentrations potentially perceived as sensory defects.

The nutritional composition of the must, particularly the availability of yeast assimilable nitrogen (YAN), plays a central role in yeast growth and fermentation efficiency (HOUTMAN et al., 2017; ROLLERO et al., 2018). Nitrogen-deficient musts are strongly associated with sluggish or stuck fermentations and favor the formation of reductive sulfur compounds, such as hydrogen sulfide (H_2S), which impart undesirable sensory characteristics to both wine and distillate (BRAMLEY et al., 2017).

Houtman et al. (2017) demonstrated that nutrient availability, especially nitrogen, significantly influences fermentation of kinetics and the aromatic profile of wine. Complementarily, Rollero et al. (2018) showed that different yeast species exhibit distinct metabolic strategies for nutrient acquisition and utilization, and that nutritional competition in mixed fermentations can substantially alter fermentation dynamics. Consequently, nutrient supplementation, particularly nitrogen addition in the form of diammonium phosphate (DAP) or complex nutrients containing amino acids, vitamins, and minerals, is a well-established practice in modern winemaking aimed at preventing problematic fermentations and optimizing the formation of desirable aroma compounds.

The initial sugar concentration of the most directly determines the final alcohol content of the wine and imposes significant metabolic challenges on yeasts, particularly under high- and very high-gravity (VHG) conditions. Zhang et

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al. (2024) demonstrated that elevated sugar concentrations exceeding 300 g/L impose severe osmotic stress on yeast cells, resulting in slower fermentations, increased glycerol production as an osmoregulatory mechanism, and a higher risk of incomplete fermentation. Under such conditions, the selection of yeast strains with high osmotic and ethanol tolerance becomes essential.

Cruz et al. (2018) evaluated the performance of different Saccharomyces cerevisiae strains under high-sugar fermentation conditions and observed prolonged lag phases, reduced cell viability, and compromised fermentation efficiency. The authors also identified temperature as an aggravating factor, showing that temperatures above 30°C, combined with high sugar concentrations, intensify cellular stress. In a subsequent study, Cruz et al. (2021) demonstrated that fed-batch fermentation constitutes an effective strategy to mitigate these effects by maintaining sugar concentrations at sublethal levels, thereby enabling the production of alcohol contents exceeding 16% (v/v) while preserving cell viability and productivity. The authors concluded that, for concentrated must, integrated management of fermentation temperature, optimized around 27 °C, and substrate feeding strategy is more effective than simple strain selection, allowing the reconciliation of high productivity with prolonged yeast viability.

Finally, it is essential to emphasize that distillate quality is intrinsically linked to the quality of the ferment from which it is derived. Undesirable congeners formed during poorly managed fermentations, such as excessive acetic acid or elevated methanol concentrations, not only compromise the sensory profile of the final product but also reduce distillation efficiency by requiring stricter cut points and resulting in lower yields. Thus, fermentation should not be regarded as a merely preparatory step, but rather as the technical and sensory foundation upon which product excellence is built. Mastery of the parameters discussed herein is therefore a fundamental prerequisite for studies aimed at optimizing distillation operations, whether in plate columns or differential systems, as proposed in this work.

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Distillation

The distillation stage separates the components based on their volatility. The most volatile compounds, such as methanol and acetaldehyde, are distilled first, in the "heads" fraction. The less volatile ones, such as higher alcohols, are distilled later in the "hearts" and "tails" fractions. The desirable part of the beverage corresponds to the intermediate fraction (hearts), which represents 80-85% of the total distilled volume, is rich in ethanol, and contains the smallest amount of secondary compounds that distill along with the water-ethanol mixture (CANCELIER, et. al., 2013).

Among the beverages made from distilled wine, the best known are Cognac and Armagnac in France, Brandy in Spain, Pisco in Peru and Chile, and Singani in Bolivia. The name "Cognac" refers to the designation of distilled wine aged in oak barrels from the Charentes region of France (RIZZON & MENEGUZZO, 2001).

Wine spirit is defined as a spirit obtained through the distillation of wine, fortified wine, or wine distillate (previously distilled to less than 86% alcohol by volume. No maturation period has been established. Brandy is a spirit made from wine spirit (distillate at <86% alcohol by volume, with a high concentration of less volatile than ethanol congeneric compounds, mainly higher alcohols and esters), which may be blended with wine distillate, rich in more volatile than ethanol congeneric compounds, mainly acetaldehyde, acetal, and ethyl acetate): DANIEL BUTRÓN-BENÍTEZ, et al., 2024.

Batch distillation is a unit operation widely used in the fine chemical, pharmaceutical, biochemical, and food industries to process small quantities of high-value-added materials. Batch distillation can be operated with a constant or variable reflux rate, and even without reflux, which constitutes differential distillation (LOPES, 2008). To obtain higher concentrations of ethanol in the distillate, a perforated plate column with reflux is used (McCABE et al. 2004). The Figure (1) consists of a diagram of a distillation unit, showing how to save energy by using the heat of the distillate to heat the wine (DOUADY et a., 2019).

Performing a differential mass balance yields Equation (1), Rayleigh's Equation; the concentration of the distillate (x_D) can be obtained by a simple matter

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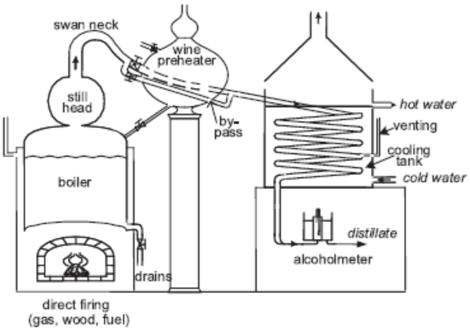
balance, Equation (2): TREYBAL, 1981.

Figure 1. Diagram of a batch distillation column (DOUADY et al., 2019).



$$\int_{W}^{F}\frac{dL}{L}=ln\frac{F}{W}=\int_{xw}^{x_F}\frac{d_x}{y-x} \quad (1)$$

$$F\cdot x_F=D\cdot x_D+W\cdot x_W \quad (2)$$

Rayleigh's model has limited accuracy compared to distillation using perforated plates, primarily due to the assumption of equilibrium occurring between the vapor and liquid phases.

F is the molar concentration charge x_F and W is the molar concentration of residual liquid with molar composition x_W. In general, in a binary system, Equations (3) to (6) apply for distillation with rectification (McCABE et al., 2004).

Total material balance

$$F=D+W \quad (3)$$

From Equations (2) and (3):

$$\frac{D}{F}=\frac{x_F-x_W}{x_D-x_W} \quad (4)$$

$$\frac{W}{F}=\frac{x_D-x_F}{x_D-x_W} \quad (5)$$

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3. Methodology

Figure 6 shows the distillation column used in the study, which contains 10 perforated plates and is programmed to operate with a reflux ratio of 4. At the base, heat is supplied by a heating system using electrical resistance. Figure 7 shows the top of the column, detailing the vapor condensation system and the reflux device.

Figure 6. View of the distillation column.

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Figure 7. Top of the column.

Figure 8 shows the refractometer (ATAGO- Model NAR-1T), with accessories: digital thermometer and thermostatic bath, with a working temperature of 21°C, used for measuring alcohol concentrations. Figure 9 shows the refractometer's adjustment curve, allowing the alcohol concentration to be obtained as a function of the refractive index.

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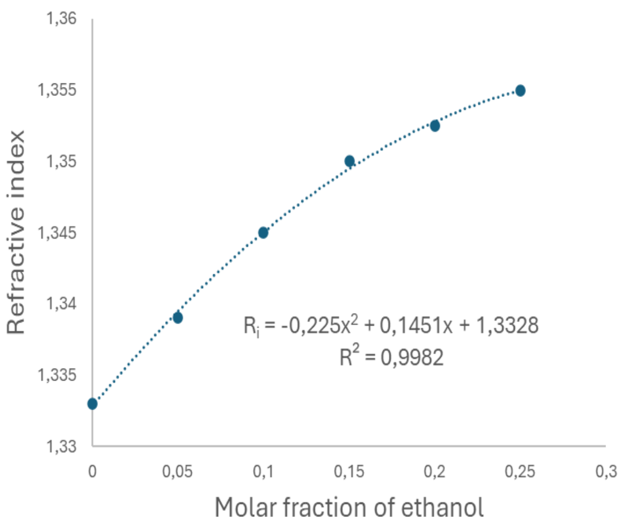
Figure 8. **View of refractometer with accessories.**

Figure 9. refractometer's adjustment curve



The amplitude of the distillate concentration depends on the liquid-vapor equilibrium, which is represented in Figure 10, for the molar fractions in the vapor and liquid phases (HARRICH et al., 2009). The experimental data were fitted to Equation (6):

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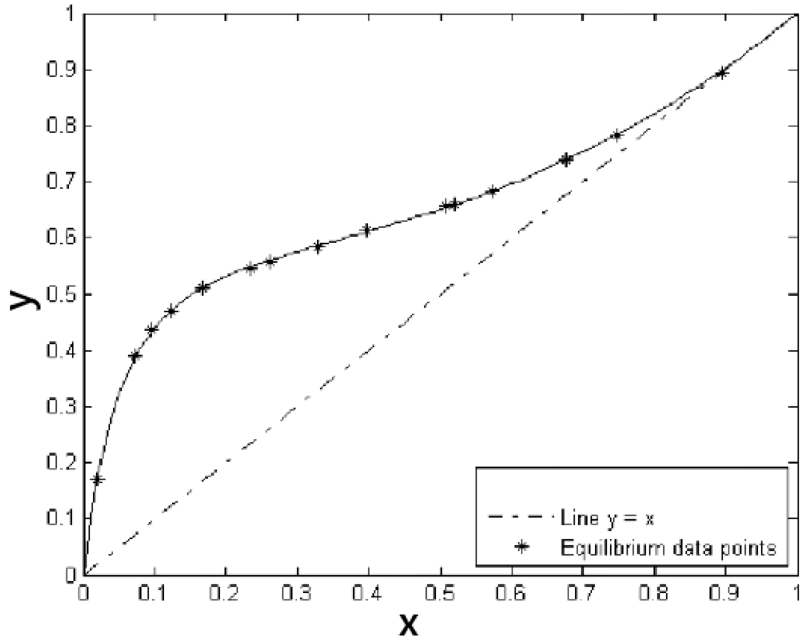
Figure 10. Equilibrium curve of the ethanol-water system. Fonte: LEAL, 2015.



$$y = -42.931x^6 + 145.08x^5 - 191.95x^4 + 126.28x^3 - 42.95x^2 + 7.4445x + 0.0203 \quad (6)$$

$R^2 = 0.998$

4. Results and Discussion

Perforated plate column distillation

The feed of fermented grapes (simulated) to the plate distillation column was 2,060.28 g. The concentration measurement with an Alcoomètre: Dujardin-Saligron - series 153, Paris, indicated 12 GL. ITEC table: ethanol concentration, 9.7% by mass; density 0.984 g/mL.

The mass of ethanol in the feed was 199.85 g; the average mole weight of the mixture was obtained using 100 g of feed as the basis for calculation. The average mole weight is calculated by dividing by the mass, expressed in moles, of the mixture's components. Then, the average mole of the mixture was quantified using a calculation basis of 100 g of the sample: 19.13 kg/kmol. Therefore, the

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amount of matter fed was 0.108 kmol; whose molar concentration was 0.04.

The mass of distillate obtained was 183.73 g. The density of the distillate was quantified by measuring the volume and mass of samples, obtaining an average density of 0.838 g/mL. The alcohol concentration was 83.6% (PERRY, 2004). The mass of ethanol in the distillate was 151.58 g. Therefore, x_D was quantified as 0.65. The amount of distilled matter was 0.00508 kmol. Therefore, W = 0.103 kmol (the mass at the end of distillation was 1,803.02 g). The result showed that approximately 5% of the molar feed was distilled.

Using the refractometer, and the curve fitted in Figure 9, the molar fraction of W was obtained as: 0.0125.

The partial mass balance for ethanol indicates that:

$$0.108\ kmol \cdot 0.040 = 0.00508\ kmol \cdot 0.65 + 0.103\ kmol \cdot 0.0125$$

$$0.00432 = 0.003302 + 0.0012875$$

$$0.00432 \simeq 0.00469$$

Deviation of 5,9%

Considering that the partial mass balance includes quantities of mixtures and concentrations, obtained using a balance with a resolution of 0.01 g and a calibrated refractometer, and that mass losses are common in distillation, such as in the evaporation of volatiles. The literature describes that deviations of 4 to 8% are common and depend on the technique and equipment used. Thus, the quantified deviation is within the range of conventional results.

A distillation with a reflux ratio (R) of 4 is considered a high to moderately high reflux operation, resulting in high product purity but with a significant increase in energy consumption. A reflux ratio of 4 means that for every 1 mole of product (distillate) withdrawn, 4 moles of condensed liquid return to the column. This requires the reboiler to vaporize much more liquid (the vapor flow rate at the top of the column is 5 times greater than the product). High reflux promotes intense contact between liquid and vapor, enriching the vapor with more volatile

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components and ensuring a product of very high purity. High reflux reduces the number of plates. Distilling with reflux 4 is a technical choice focused on purity, sacrificing energy efficiency. Increasing the reflux to 4 will consume more steam in the reboiler and cooling water in the condenser (McCABE et al., 2007). The distillation column supplier Normschliff – Distillation plants – Germany, reports efficiencies between 60 and 65%, however, this depends on the distilled material and the flow rates of the liquid and vapor phases. In the current study, simulating an overall efficiency of 60%, the theoretical number of plates would be 6.

Differential distillation

Differential distillation performance is different. Performing a simulation on the distillation of 5% of the feed, expressed in mol.

Using Equation (1) and data from Figure 10, data was obtained for integrating the equation (Figure 11):

$$\int_{W}^{F} \frac{dL}{L} = ln\frac{0.1080}{0.1026} = 0.051 = \int_{x_W}^{0{,}04} (0{,}8089x^{-0.578}) \cdot dx$$

Integrating:

$$0.051 = 0.8089 \cdot \frac{[0.04^{0.422} - x_W{}^{0.422}]}{0.422}$$

Then: $x_W = 0.031$

From the molar mass balance: $x_D = 0.211$

Compared with distillation in a perforated plate column:

$$\frac{x_{D\,Column\ of\ plates}}{x_{D\,Differential\ column}} = \frac{0.650}{0.211} = 3.08$$

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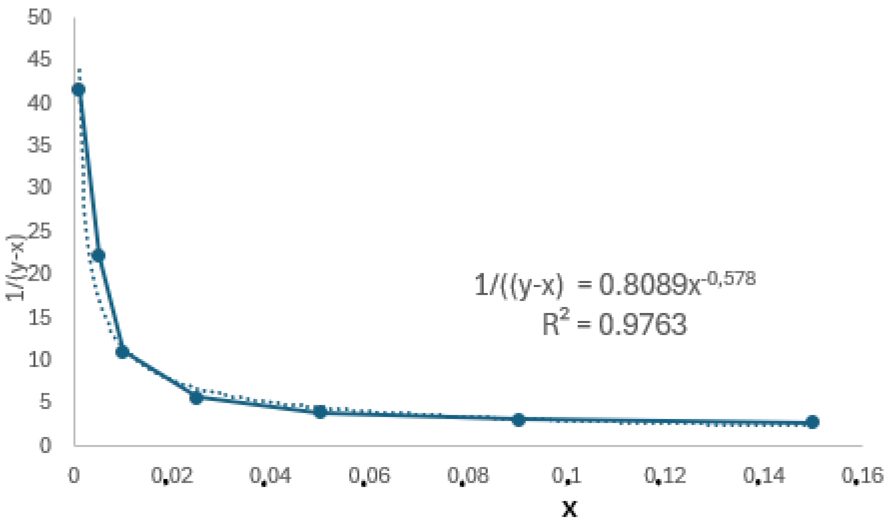
Figure 11. Curve fitting for integration



Therefore, when using a perforated plate column with reflux, the same quantity of distillate is obtained, resulting in a concentration three times higher. Consequently, the alcoholic concentration of the bottom product from the differential distillation will be significantly higher.

5. Conclusion

The study demonstrates the results obtained from the distillation of wine in a perforated plate distillation column, showing that the distillate concentration, compared to differential distillation, is approximately three times higher when the same quantity of distillate is obtained. The quality of the distillate, however, depends on instrumental measurements of the distillate. The designations of Cognac aged in oak barrels or Brandy with concentrations of components other than ethanol depend on chromatographic assays of distillates. Therefore, the topic does not end here, and expanded studies should be developed using distilled products from fermented wine.

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