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# Dynamic Simulation of Batch Distillation in the Cognac Production Process

TESI DI LAUREA MAGISTRALE IN  
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# Abstract

The growing influence of Digital Twin (DT) technology and advanced simulation software is transforming traditional food and beverage industries through dynamic simulation tools, data-driven decision-making, and opportunities for process optimization.

This thesis investigates the dynamic simulation of the distillation process in Cognac production using the DYNOSIM® software. The main goal is to digitalize traditional technology by modeling the Charentais alambic still, focusing on accurately reproducing the second distillation stage.

Using DYNOSIM® tools, a detailed process flowsheet was developed, covering heating, reflux, product separation, and shutdown phases. Basic control strategies were implemented to manage key process variables. Reliable models were created for each unit, ensuring that no numerical errors occur throughout the simulation, and using the NRTL (Non-Random Two-Liquid) thermodynamic model.

According to classical technology, 2,500 liters of a water-ethanol mixture representing low wine were virtually distilled. The output was separated into four fractions based on alcohol content. Simulation results were presented in graphs and tables illustrating key stages and system dynamics.

DYNOSIM® successfully replicated the traditional distillation process, capturing the system's dynamic behavior and showing a strong correlation with real-world patterns, including the expected volatility sequence and compound-specific trends.

A special focus was placed on the behavior of five key volatile compounds—acetaldehyde, ethyl acetate, methanol, isoamyl alcohol, and furfural—selected for their sensory impact, relevance to product quality and varying volatilities. Their distribution across fractions was analyzed, and compared with literature data, confirming the simulation's ability to replicate complex, time-dependent operations.

Due to lower costs and shorter time required, simulation proves a competitive alternative to physical experiments. As distillation remains an energy-intensive operation in the industry, the study helps to find ways for optimization, improving efficiency, sustainability, and product quality for classical technology. Finally, the thesis highlights the potential for expanding the model with additional components, control strategies, and energy-saving techniques, thereby demonstrating the feasibility of future digital transformation in the spirits industry.

**Key-words:** DYNOSIM®, Cognac; batch distillation; simulation; volatile aroma compounds.

## Abstract in italiano

L'influenza crescente della tecnologia del Digital Twin (DT) e dei software di simulazione avanzata sta trasformando le industrie tradizionali alimentari e delle bevande, grazie all'integrazione di strumenti di simulazione dinamica, decisioni basate sui dati e nuove opportunità di ottimizzazione dei processi.

Questa tesi analizza la simulazione dinamica del processo di distillazione nella produzione del Cognac utilizzando il software DYNOSIM®. L'obiettivo principale è digitalizzare la tecnologia tradizionale modellando l'alambicco Charentais, con particolare attenzione alla riproduzione accurata della seconda fase di distillazione.

Attraverso il software DYNOSIM® è stato sviluppato un diagramma di flusso dettagliato del processo, includendo le fasi di riscaldamento, riflusso, separazione del prodotto e arresto. Sono state implementate strategie di controllo di base per gestire le variabili di processo principali. Modelli affidabili sono stati creati per ogni unità e operazione fisica, evitando errori numerici durante la simulazione, grazie all'utilizzo del modello termodinamico NRTL (Non-Random Two-Liquid).

Secondo la tecnologia classica, sono stati distillati in modo condizionale 2.500 litri di una miscela acqua-etanolo rappresentativa del "brouillis". Il prodotto ottenuto è stato suddiviso in quattro frazioni distinte in base al contenuto alcolico. I risultati della simulazione sono stati presentati attraverso grafici e tabelle che illustrano le fasi principali e la dinamica del sistema.

DYNOSIM® ha riprodotto con successo il processo di distillazione tradizionale, catturando il comportamento dinamico del sistema e mostrando una forte correlazione con i modelli reali, inclusa la sequenza di volatilità attesa e le tendenze specifiche dei composti.

Particolare attenzione è stata dedicata a cinque composti volatili chiave—acetaldeide, acetato di etile, metanolo, alcol isoamilico e furfurale—scelti

per il loro impatto sensoriale, rilevanza nella qualità del prodotto e diversa volatilità. La loro distribuzione tra le frazioni è stata analizzata e confrontata con dati presenti in letteratura, confermando la capacità della simulazione di riprodurre operazioni complesse e dipendenti dal tempo.

Grazie ai costi ridotti e ai tempi brevi di analisi, la simulazione si dimostra un'alternativa competitiva agli esperimenti fisici. Poiché la distillazione è ancora oggi una delle operazioni più energivore, lo studio contribuisce all'identificazione di strategie di ottimizzazione, migliorando efficienza, sostenibilità e qualità del prodotto nella tecnologia tradizionale. Infine, la tesi evidenzia il potenziale di espansione del modello attraverso l'integrazione di nuovi componenti, strategie di controllo avanzate e tecniche di risparmio energetico, dimostrando la fattibilità della trasformazione digitale nel settore degli alcolici.

**Parole chiave:** DYNOSIM®, Cognac; distillazione discontinua; simulazione; composti aromatici volatili.

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# 1 Introduction

## 1.1. Thesis overview

This thesis is divided into five main chapters, each focusing on an important part of the research. The first chapter, Introduction, gives a general overview of current knowledge in areas such as distillation and process simulation, with an emphasis on how these methods are used in the production of alcoholic beverages. It also explains the aims of this thesis, highlighting the value of bringing traditional technologies into the digital age by using modern simulation software.

The Theoretical Background reveals the fundamental principles of distillation concerning the need to adapt food technologies to changing trends in modern industry and emerging modern technologies such as Industry 4.0, as well as the mathematical models and technological processes used to perform operations. This section is crucial for understanding the complex behavior of the distillation process, especially in dynamic states.

The Methods and Tools chapter explains how the selected simulation software is used and which specific tools help to build an effective working simulation. In addition, it describes the steps involved in setting up the dynamic simulation — from thermodynamic models and creating the process flowsheet to implementing control strategies, and comments in detail the carried-out process. The scenarios used for the simulation are presented, which give an idea of why a particular choice is made.

The Results and Discussion chapter presents simulation results analyzing the dynamic behavior of a batch distillation column at various stages of the process. Key performance indicators such as intermediate and final product concentration, time, and performance parameters are presented as graphs and commented in detail. Finally, the Conclusion summarizes the main contributions of the thesis. It also outlines potential future steps to improve the simulation model and its applications.

## 1.2. State of the Art

### 1.2.1. Distillation

Distillation is one of the oldest and most fundamental separation processes, that exploits differences in volatility between components in a liquid mixture, relying on vapor-liquid equilibrium (VLE) to achieve effective separation. Due to density differences between vapor and liquid phases, lower-boiling components concentrate in the rising vapor, while higher-boiling components remain in the descending liquid. The separation efficiency is enhanced by countercurrent flow in the distillation column, resulting in a vapor phase progressively enriched in light components and a liquid phase enriched in heavier components as they move through the column [1].

The lighter components are then converted into liquid in the condenser. A heat exchanger located at the top of the column condenses the vapor, rich in the more volatile components. Part of that condensed vapor is extracted as product and part is returned to the column to get into contact with the rising steam, and this process is known as reflux.

While the use of distillation dates back in recorded history to about 50 B.C., the first truly industrial exploitation of this separation process did not occur until the 12th century when it was used in pharmacy and the production of alcoholic beverages. Today, distillation is a common and widely used process in many industries such as petroleum, chemical, petrochemical, pharmaceutical, and especially in food production — mainly in the making of alcoholic beverages.

### 1.2.2. Basic Principles of Distillation

When a mixture of two or more liquids is heated and boiled, the vapor has a different composition than the liquid. For example, if a 10% mixture of ethanol in water is boiled, the vapor will contain over 50% ethanol. The

vapor can be condensed and boiled again, which will result in an even higher concentration of ethanol. Distillation operates on this principle [2].

Vapor–liquid equilibrium (VLE) data are fundamental for the design and optimization of distillation processes, it describes the distribution of components between the vapor and liquid phases at equilibrium, which depends on factors such as temperature, pressure, and mixture composition. This equilibrium is governed by mathematical models known as equations of state; relationships that connect these variables and help predict the system’s behavior under various conditions. By applying VLE principles along with appropriate thermodynamic models, engineers can design more efficient distillation systems that achieve the desired separation while minimizing energy consumption [1].

Vapor-liquid equilibrium calculations in binary mixtures consist of determining two variables of the set temperature, pressure, liquid phase concentration, and vapor phase concentration ( $T, P, x, y$ ), when the other two are known. For a vapor-liquid mixture at equilibrium conditions, the temperature and the pressure are the same in both phases, and the remaining variables are defined by the material balance and the “fundamental equation of phase equilibrium.” For engineering purposes, this fundamental equation is conveniently expressed in terms of the fugacity of each component in the different phases [3].

For ideal mixtures, equilibrium behavior can be described using Raoult’s law. However, many real systems, such as the water–ethanol mixture, exhibit non-ideal behavior.

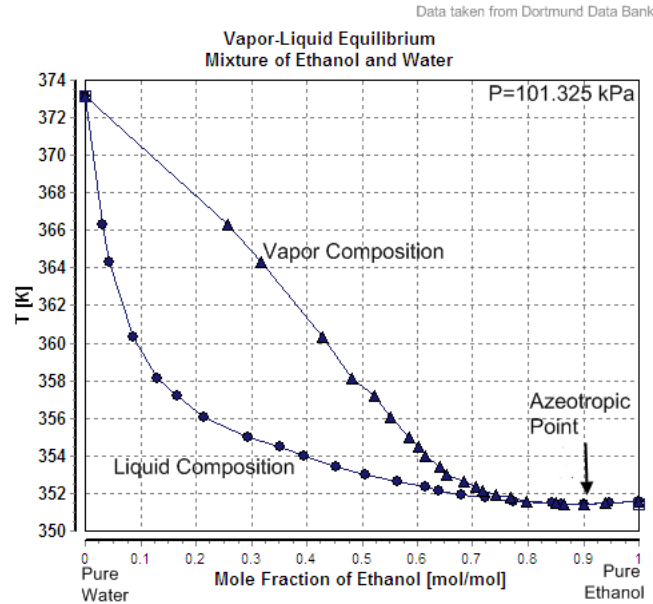


Figure 1 Vapor-liquid equilibrium for water-ethanol mixture

As shown in the graph at Figure 1, water-ethanol displays a particular behavior that differs from standard binary mixture equilibrium. In fact, water and ethanol form an azeotropic mixture at approximately 89% ethanol and 11% water on a mol/mol basis [4].

However, in the context of this study, where distillation is modeled for a mixture resembling wine and its distillation products, the azeotrope does not represent a critical issue, as neither the feed nor the products reach ethanol concentrations that high [5].

### 1.2.3. Continuous and Batch Distillation

There are two main kinds of distillations: continuous and batch. In continuous distillation columns, there is a constant feed, a lighter product is constantly retrieved from the top (distillate) and a heavier one from the bottom (residue). Continuous distillation operates without interruption, maintaining a steady input of feed and output of products, making it ideal for large-scale industries like petrochemicals and high-volume beverage production.

Batch distillation, in contrast, processes finite quantities of feed per cycle, offering greater flexibility and control over product composition, which is particularly beneficial for specialty chemicals, pharmaceuticals, and traditional spirit production. While continuous distillation is more efficient and automated, requiring high initial investment, batch distillation is more adaptable to variable feedstocks and allows for precise control of flavors and aromas in alcoholic beverages.

In batch distillation columns the mixture to separate is charged at the beginning in the reboiler. The operation is then started heating the blend. The lighter component is first evaporated and continuously accumulated on the top, while the heavier elements are concentrated at the bottom. Since the amount and concentration of distillate and residue changes over time, the process is unsteady-state. Batch distillation is preferred when small amounts of mixtures are treated or when there are big changes in the feed composition. Furthermore, it requires a lower capital cost. Batch distillation is also the standard choice when there are seasonal operations or different production campaigns [2].

### 1.3. Process Simulation

Process simulation is a way to describe chemical processes through mathematical models, which can then be used to obtain information about their performance. The application of simulation techniques in chemical engineering started gaining traction in the mid-1950s, and the first simulation software was published in 1958. In the mid 1960's the most popular current structure for process simulation, namely, the modular program was developed. This method allows a chemical process to be represented by linking together a set of standard mathematical modules into a flexible network. In recent years, process simulation has become an accepted tool for the design and understanding of chemical processes and almost a requirement in chemical engineering education. The rising interest in this area can be seen in the increasing number of process simulation programs which are offered for sale to the chemical industry, as well as the rapid growth of research publications in this field [6].

### 1.3.1. Modeling

Modeling batch distillation processes is particularly challenging due to their complex and dynamic behavior. Start-up involves a transition from the initial state to a pseudo-steady state, where the desired product composition is achieved. Since these dynamic transitions are always considered as non-productive periods, many researches have been performed to minimize the start-up time, energy use, and waste generation.

The mathematical model for batch distillation is formulated as a set of differential and algebraic equations (DAE). Ordinary differential equations (ODE) represent energy balances, total and component mass balances. Algebraic equations are composed of constitutive equations such as vapor liquid equilibrium relationships, summation equations, physical property estimations, etc. In order to reduce the complexity of the model, the following typical assumptions were made:

1. Perfect mixing of vapor bubbles and of liquid phase;
2. Equilibrium relationship between liquid and vapor with possible introduction of the Murphree efficiency;
3. Negligible vapor hold-up compared to liquid one;
4. Constant volume of the liquid hold-up once filling up is completed.

$$\frac{dY}{dt} = F(Y, X, P, t) \quad (1)$$

$$X = S(Y, X, D) \quad (2)$$

$$P_1 = H_1(X, D) \quad (3)$$

$$P_2 = H_1(Y, D) \quad (4)$$

In the above equations:

- The ordinary differential equation (ODE) (1) represents the balance equations;
- Equation (2) represents the implicit algebraic equations resulting from thermodynamic property and phase equilibrium calculations;
- Equations (3) and (4) are explicit algebraic equations.

As for the variables:

- Y is the vector of differential variables;
- X is the vector of implicit algebraic variables;
- P is the vector of explicit algebraic variables;
- D is the vector of specified (design) variables [7], [8].

The simulation is created by the user through a graphical interface that enables the construction of a flowsheet by connecting equipment models with material, thermal, and mechanical streams. This setup provides a clear, logical representation of the plant's configuration for simulation purposes. From an industrial perspective, the simplicity and flexibility of such models are particularly beneficial for optimizing batch operations, especially during the transition from the initial cold state to stable operation with total reflux. This approach offers greater accuracy and efficiency compared to traditional models, making it especially valuable in time-dependent optimization problems [9].

### 1.3.2. Modeling Wine Distillation Process

Simulating batch distillation is more complex than steady-state processes due to its dynamic nature [10]. Although advanced simulators are powerful, they are not widely used in food processing, primarily due to limited data on specific compounds and the complexity of these processes [11].

Thermodynamically, wine is a complex, non-ideal colloidal suspension primarily composed of water and ethanol, with minor volatile compounds that significantly impact the quality of the distillate. During distillation, the goal is to concentrate ethanol and desirable aromas while minimizing off-flavors.

Since water and ethanol make up over 96% of the distillate, properties like density, enthalpy, and boiling point are typically derived from water-ethanol binary data, ignoring minor components and heat effects. Accurately modeling a batch distillation process makes it possible to determine the exact moment when given substance is coming in the

distillate at a high concentration, as well as how long that concentration remains high [10].

Classical thermodynamic models for these mixtures often rely on numerous binary parameters derived from experimental data. Among the available liquid-phase models for atmospheric pressure, the NRTL model provides the best results, flexibility and sufficient accuracy in modeling the treatment modes of water + congener and ethanol + congener mixtures [12] [13].

## 1.4. Digital Twin

The growing need for accurate modeling corresponds with the shift towards Industry 4.0, which focuses on accelerating the manufacturing with lower cost and higher productivity by integrating digital technologies such as the Internet of Things (IoT), cyber-physical systems (CPS), and cloud computing. Industry 4.0 uses artificial intelligence for data-driven decision making, autonomous problem solving, and continuous learning to optimize processes over time [14].

A Digital Twin (DT) is a key technology in Industry 4.0 that connects physical systems with their virtual counterparts using models, sensors, data, and software. It functions as a dynamic model that continuously adapts to operational changes based on real-time data, enabling monitoring and performance analysis, helping to track productivity, predict future behavior, and support decision-making in production processes [15].

Typical commercial modeling software includes the following components: component database, thermodynamic model database, flowsheet builder, unit operation model database, and flowsheet solver.

Figure 2 shows a list of sequential steps in solving the modeling problem [16].

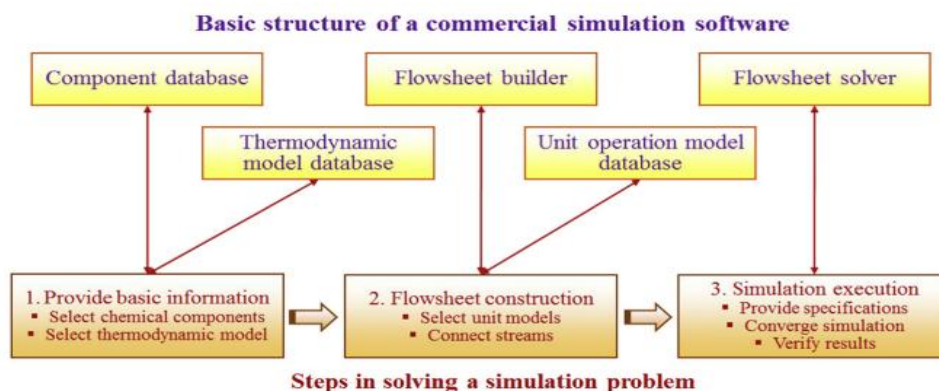


Figure 2 Basic structure of a commercial simulation software and sequence in solving a simulation model

### 1.4.1. Dynamic Simulator

The history of commercial process simulation software was dated in the 1960s. With the development of simulation software, at the beginning of the 80s the modeling and simulation of distillation processes has become faster and easier.

Commercial simulation tools like AVEVA™ Dynamic Simulation (DYNSIM®) have been developed to address these needs, providing advanced dynamic modeling capabilities throughout the entire lifecycle of a process plant. DYNSIM® enables detailed simulations of transient operations, allowing engineers to assess equipment performance, test control strategies, and optimize design for cost reduction and safety. It is particularly effective in modeling time-dependent processes, such as start-up, shutdown, and other transient states in batch distillation [17].

In the food sector, there has not yet been a general simulation program applicable to any wine distillation process, although several specific applications for wine distillation have been reported [13], [18].

The wine industry may be the most complex within the food industries panorama. Winemaking still has a high degree of craftsmanship; it relies on the experience of great winemakers rather than on a standardized process. Data-driven food-manufacturing systems ensure cost reduction,

increased efficiency, and product quality. These elements come together in an effective cycle of digital transformations focused on sustainability [19].

In particular, this work aims to develop a simulation of the Cognac production process with a focus on the second distillation to produce final distillate for aging. This performs a single unit operation in a batch distillation apparatus simulated in software DYNOSIM®, and by giving attention to the different steps, from the actual beginning to the end of the operations.

Since it is rare to find simulation results in this special field in the literature, the purpose of this work is to fill this gap and test the feasibility of using software capabilities in such a delicate and conservative field as winemaking, to digitalize classical distillation technology by applying modern simulation tools.

## 1.5. Thesis Objectives

Simulation provides a powerful tool for studying and analyzing processes without the need for conducting physical experiments. It is increasingly evident that the precision of dynamic models integrated into commercial software such as DYNOSIM® has improved significantly over the years. This growing accuracy allows to bring the simulation results closer to the results of real experimental tests. Thanks to this it makes possible to explore delicate processing operations that were traditionally considered untouchable or too sensitive for external intervention. Due to the lower costs and shorter time required to analyze each process, simulation becomes a highly efficient and competitive alternative to physical experiments, especially in the early stages of process development and design. Given that distillation is one of the most energy-intensive operations in industry, the study can help to find ways for optimization, improving efficiency, sustainability, and product quality for classical technology through advanced modeling techniques [20], [21].

The objective of this work is to simulate the second distillation step in the Cognac production process, focusing on producing the final distillate

intended for aging. The simulation is conducted using DYNOSIM®, replicating each phase of the batch distillation operation from start to finish. Given the limited availability of simulation studies in this specific area, this work seeks to bridge that gap and explore the potential of using advanced simulation software to digitalize traditional distillation techniques in the highly traditional field of winemaking.

Determination of flavor volatile compounds in water-ethanol solutions is usually performed by time-consuming and costly standard methods such as chromatography or the Gillespie dynamic recirculation method. This study tested possibility of capturing the dynamics of volatile substances, presented in ultra-small concentrations during distillation process, by the DYNOSIM® software.

## 2 Theoretical Background

### 2.1. Brandy and Cognac

Since the Middle Ages, distillation has been used to produce alcoholic beverages from fermented plant materials rich in carbohydrates. Across different regions, unique ingredients and traditions have shaped a diverse range of spirits: in Scotland, whiskey is made from barley, while in the Mediterranean, wine serves as the base for brandy, in North America produce bourbon from corn and rye, Mexico distills agave into tequila, Scandinavia relies on potatoes for spirits, the Caribbean transforms sugarcane into rum, and many other well-known distilled beverages, including vodka, gin, pisco, cachaça, absinthe, calvados, slivovitz, and raki, each reflecting the cultural heritage and raw materials of its region [22].

The name brandy, used alone, generally refers to the grape product; brandies made from wines or fermented mashes of other fruits are commonly identified by the specific fruit name. The history of brandy production dates back to the 12th century when it was exclusively made by apothecaries and used as medicine. At that time, distillation was carried out on a small scale, and the quality of the product depended heavily on local practices. It was not until the 16th century that brandy production expanded, particularly in France, when distillation was officially authorized by French authorities. Today, brandy is produced worldwide, yet France, with its renowned Cognac, remains the global leader in quality and reputation.

According to modern EU regulations (EEC, 1989) brandy or *weinbrand* '...is a spirit drink, produced from wine spirit, matured for at least one year in oak receptacles or for at least six months in oak casks [...], containing a quantity of volatile substances equal to or exceeding 125 grams per hectoliter of 100% vol. alcohol, and derived exclusively from the distillation or redistillation of the raw materials used, having a maximum

methanol content of 200 grams per hectoliter of 100% vol. alcohol. The minimum alcoholic strength by volume of brandy or *weinbrand* shall be 36%' [23].

Brandy and Cognac both belong to the broader family of wine-based spirits, and Cognac is a special case of brandy. However, they differ significantly in their production methods, appellations, taste profiles, and international recognition. Cognac, unlike generic brandy, must be produced in a specific region of France, centered around the town of Cognac, and must adhere to strict regulations set by the National Interprofessional Bureau of Cognac (BNIC). These regulations ensure the authenticity and quality of the product, including controls on grape varieties, distillation methods, and aging requirements. In contrast, brandy is a broader category that can be produced in various wine-growing regions worldwide, without the same stringent restrictions.

Brandy, on the other hand, is made from a wider variety of grapes, including both red and white varieties. In some cases, even fruit brandies exist. The distillation process for brandy is often simpler, typically involving a single distillation, which can result in a more straightforward flavor profile. Additionally, aging requirements for brandy vary significantly and depend on the production methods, local traditions, and intended final product characteristics.

The production process of Cognac is another defining characteristic. Only specific white grape varieties, such as Ugni Blanc (also known in Italy as Trebbiano), Colombard, and Folle Blanche, are permitted for cognac production. These grapes are fermented into a low-alcohol white wine, that is not the best wine to drink, it should not be intense in terms of aromas or taste, it contains between 8 and 10 % alcohol, crisp acidity, low sugars (due to the fact that during heating, the pentoses give rise to the formation of furfural (burnt) character, and little or no sulfur dioxide added. The methanol content of distilled spirits is the subject of strict regulation. The wine is double distilled in traditional copper pot still, called Charentais alambic. This double distillation process enhances aromatic complexity and refines the final spirit. Furthermore, Cognac

must be aged for a minimum of two years in French oak barrels, contributing to its depth, smoothness, and unique flavor profile [21], [24], [25].

Copper is used for a reason; it offers the following advantages: it is malleable; it is a good conductor of heat; it resists corrosion from fire and from wine; it reacts with wine constituents such as sulfur components and fatty acids (this property is always favorable for the Cognac or brandy quality); thus, it is a catalyst for favorable reactions between wine components [26]

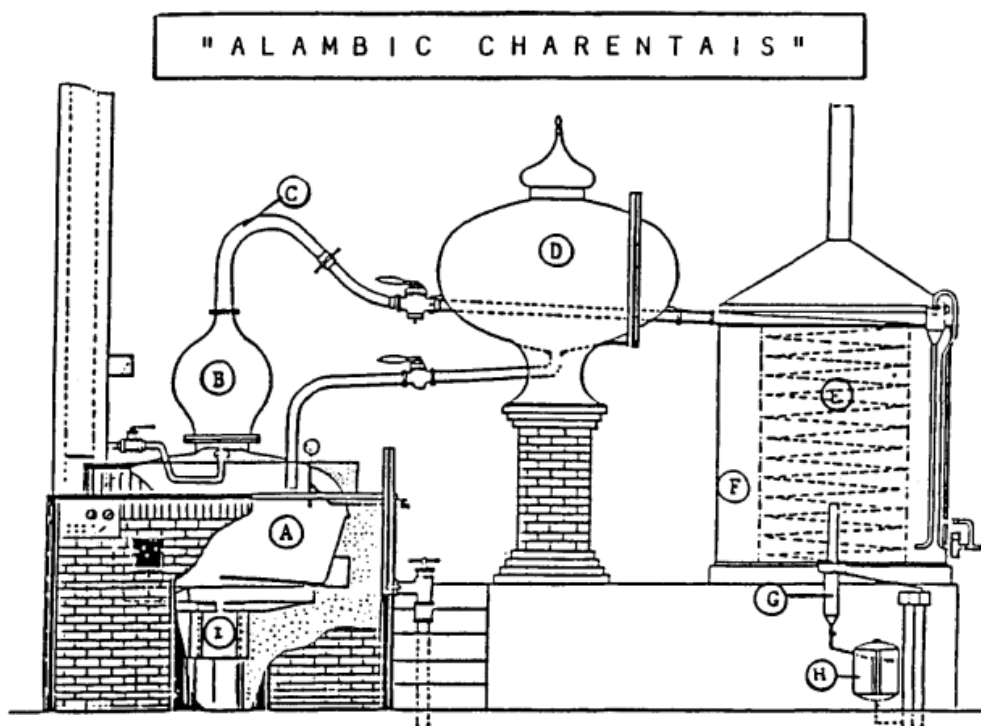


Figure 3 Diagram of the Cognac still- alambic: A standard alambic is 2500 liters capacity. (A) copper boiler (*chaudiere*); (B) hat (*chapeau*); (C) swan's neck (*col de cygne*); (D) preheater (*chauffe-vin*); (E) coil (*serpentin*); (F) condenser (*condenseur, pipe*); (G)

Hat (*chapeau*) is a part of the alambic located directly above the boiler. The volume of the hat is approximately 10% to 12% of the capacity of the boiler, depending on the specifications required by the distiller. The shape and the volume of the *chapeau* determine the concentration, selection, and separation of the different volatile components. This selection process

occurs when volatile compounds partly condense in the *chapeau* and fall back down into the boiler, where they must be re-distilled upward. This phenomenon is called the reflux process [26].

Copper alambics have been traditionally used for hundreds of years to produce spirits. Batch distillation is crucial to producing high-quality distilled spirits because the desired aromas can be selectively recovered by adequately managing the cuts (head, heart and tail) and the operating variables (reboiler heating and reflux rate) [27].

### 2.1.1. Distillation Process. Classical Technology.

The process, known as Charentaise distillation, is carried out in copper stills with a maximum load capacity of 25 hL (2.5 m<sup>3</sup>) and heated by a direct flame. Alambic stills have no trays or appreciable reflux and require multiple distillations to achieve high-proof spirits, so two successive distillations are required to produce freshly pressed Cognac containing approximately 70% ethanol: wine distillation (WD) and *brouillis* distillation (BD).

As an example of variation of the alcohol content in double distillation: if the wine is at 10% alcohol/volume, at first distillation the distillate is cut into three fractions: heads, heart (*brouillis*) and tails. The alcohol content of the distillate is around 60% alcohol/volume in the first fraction and it reaches 0% alcohol/volume at the end of the first distillation. Heads and tails are re-distilled with the succeeding batch of wine.

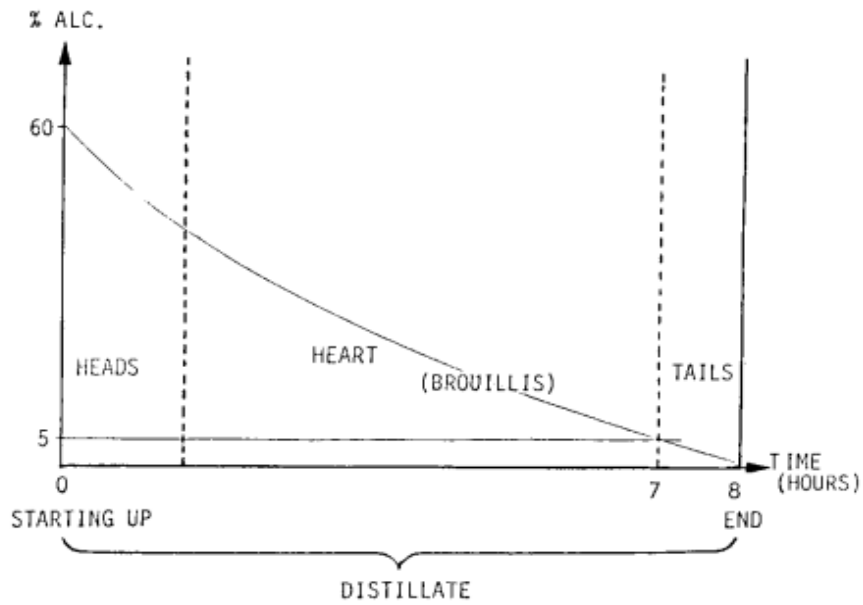


Figure 4 First distillation (wine)

The *brouillis*, which contains about 28-30% ethanol (alc/vol.), is used for the second distillation, also called "*bonne chauffe*". In second distillation, the distillate is cut into four fractions: heads, heart 1, or future Cognac itself, heart 2 or *secondes* and tails. In the first fraction, the alcohol content of the distillate is around 80% alcohol/volume and reaches 0% alcohol/volume at the end of the second distillation. Heads and tails are re-distilled with the succeeding batch of wine. *Secondes* are re-distilled with *brouillis*.

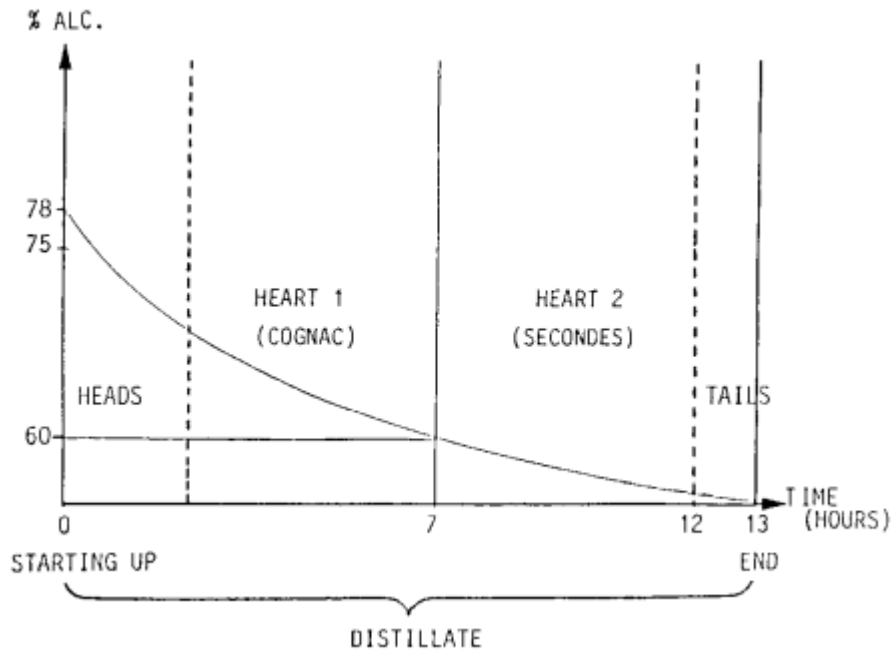


Figure 5 Second distillation (*brouillis*)

The entire and most detailed scheme is shown in the Figure 6.

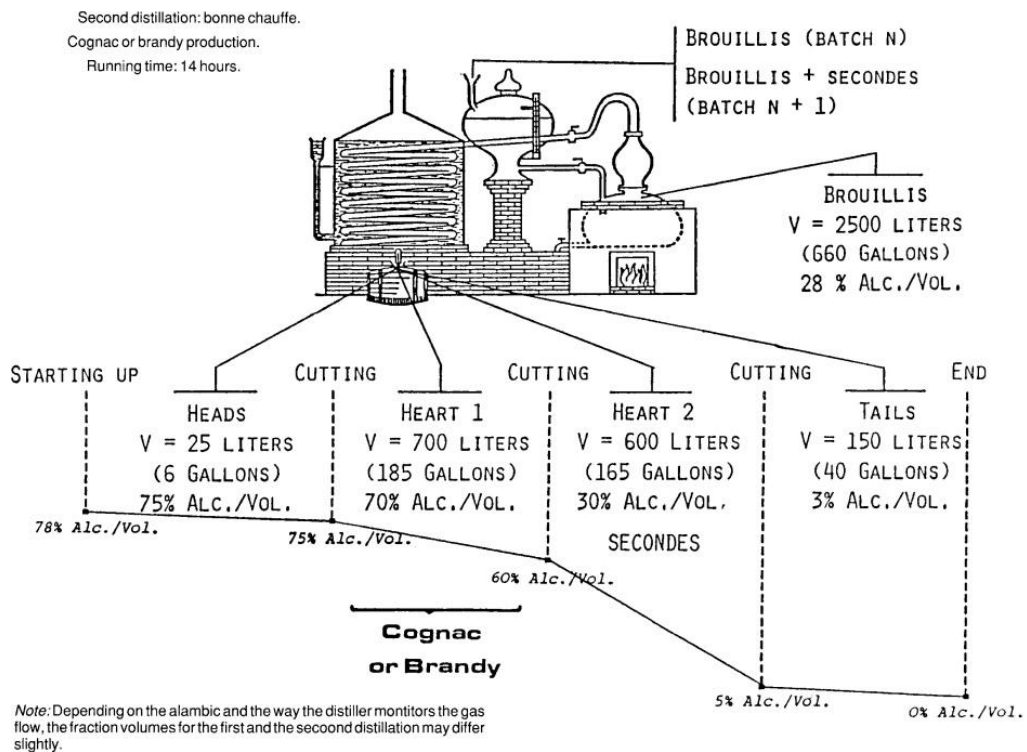


Figure 6 Scheme of the second distillation (*bonne chauffe*)

## 2.2. Volatile Aromatic Compounds in Cognac

Alcoholic beverages chemically consist of water and ethanol (representing 97 to 99% of the volume), and a wide variety (more than 500) [24] of volatile compounds at low concentrations which define the quality, flavor, and aroma of the spirit. Generally intermediate compounds are less volatile than ethanol at the beginning of distillation and gradually increase in volatility as the ethanol content in the boiler decreases. As a result, they are extracted throughout the entire distillation, being mostly present in the *brouillis* of the WD and in the heart and *second* of the BD. These flavoring compounds termed “congeners” belong to different chemical families including alcohols, esters, aldehydes, acids and terpenes. They derive from raw materials, during fermentation by yeasts, and are generated during the production process whose conditions vary according to the desired spirit.

In the production of distilled beverages, the flavor substances determine the quality of brandies, despite their extremely low concentrations ranging from a few  $\mu\text{g/L}$  to several  $\text{mg/L}$ . The characteristics of a spirit beverage largely depend not only on the nature and amount of the compounds, but also, to a lesser extent, on its ethanol content [28].

The concentration of aroma-active substances in alcoholic distilled beverages is determined by the thermodynamic processes of evaporation and condensation that occur during distillation [20].

The goal and major challenge of distillation lies in maximizing the extraction of components with pleasant aromas in the freshly distilled Cognac while limiting the presence of undesired compounds. To help achieve this goal, the distillate is often divided into different fractions in an operation known as cutting. Cutting time might be determined by the cumulated volume of distillate collected, by the ethanol concentration of the distillate measured with portable alcoholmeters or, as is most often the case, from an empirical evaluation of the distillate aroma. Four fractions are typically collected in the BD: the head, the heart, the *second* and the tail, in addition to the residue. The key fraction resulting from the process

is the heart, which corresponds to the freshly distilled Cognac that contains up to 73.7 % v/v ethanol before being stored in oak barrels for aging for at least two years.

Although cutting might help to limit the presence of some undesired compounds in the distillate, the fractions other than the *brouillis* and heart are not discarded, as they contain significant amounts of ethanol and aroma compounds that would otherwise be lost. Instead, they undergo a recycling operation, in which the heads, tails and *secondes* are redistilled in subsequent batches. Typically, heads and tails are mixed with the wine in the feeds of ensuing WDs and the *second* is blended with a new batch of *brouillis* for subsequent BDs.

Despite its importance the current scientific knowledge on the composition of freshly distilled Cognac is rather limited. As a result, the set of complex operations it entails still rely heavily on empirical knowledge and traditional practices, which lead to high variability.

Not all volatile compounds significantly contribute to overall aroma. To contribute to the scent, its concentration must exceed its odor perception threshold — the minimum level at which its smell becomes noticeable. This is measured by the odor activity values (OAV), calculated by dividing the compound's concentration by its odor threshold. Since distilled spirits typically contain around 40% ethanol, only perception thresholds reported at similar ethanol levels are considered for evaluating OAVs [29].

## 2.3. Volatile Compounds in the Focus of Our Research

Acetaldehyde, ethyl acetate, methanol, isoamyl alcohol and furfural are chosen for monitoring because they are the compounds in highest concentrations in the distillate and have a noticeable impact on the final spirits.

### 2.3.1. Acetaldehyde

Acetaldehydes are primarily formed during alcoholic fermentation. At low concentrations, they can give pleasant fruity, green, grassy notes to the beverage. However, when present in higher amounts, they cause a pungent smell, producing poor-quality distillates. With a boiling point of just 20.8 °C, acetaldehydes are among the most volatile compounds in the distillation process [22], [30], [31].

### 2.3.2. Ethyl Acetate

Ethyl acetate is the most abundant ester found in alcoholic beverages, produced by yeast during fermentation. At low concentrations (up to 200 mg/L), it adds pleasant fruity, pineapple and floral notes [22].

However, at higher levels, it can give a sharp, solvent-like aroma reminiscent of nail polish or glue [30]. The boiling point is 77 °C [31], [32].

### 2.3.3. Methanol

Methanol is the only alcohol more volatile than ethanol, and its boiling point is low (64.7 °C). It is subject to restrictions due to its high toxicity, it is a health hazard, and the content of methanol in distilled alcoholic beverages is strictly regulated. However, in the production of brandy, the methanol level is usually not a major problem, as the Ugni Blanc grape, the main variety used, naturally produces small amounts of methanol. Nevertheless, maintaining methanol concentrations close to the regulatory limit may indeed be desirable, since in small quantities it is considered a positive flavor component [30], [33].

### 2.3.4. Isoamyl alcohol

Fusel alcohols compose the largest group of aroma compounds in alcoholic beverages. Isoamyl alcohol (3-methyl-1-butanol) is the main fusel alcohol synthesized during fermentation by yeast. While it contributes

spicy, solvent-like, and pungent notes, its “damp cloth” aroma is generally considered undesirable in spirit production.[22], [31].

Boiling temperature is 132°C [31].

### 2.3.5. Furfural

Furfural is an aldehyde generated by the dehydration of pentoses, and occurs during distillation involving Maillard reactions. It gives an almond-like, sometimes smoky aroma. Its boiling temperature is much higher than water and all the water-ethanol mixture, 161°C [24].

To summarize, the above-mentioned volatile compounds – methanol, ethyl acetate, acetaldehyde, and isoamyl alcohol – were chosen for their distinct sensory characteristics and varying volatilities, which play a crucial role in the overall quality and profile of distilled spirits. The objective of this study is to see if the DT can effectively reproduce how these compounds behave during distillation, based on data from previous physical experiments. If the simulator can effectively capture these patterns, it could become a practical tool for optimizing the distillation process, reducing resource consumption, and maintaining product quality without extensive physical testing.

# 3 Methods and Tools

This section provides a brief description of the DYNsIM® software, the user interface, and the main functions used in the work.

## 3.1. DYNsIM® Dynamic Simulation

DYNsIM® organizes the process into a "Simulation" file that can include "Flowsheets" (FS) representing different plant sections. Since only one specific process is reproduced, one flowsheet is sufficient for this study. It serves as both the workspace for modeling the desired simulations and an operating environment in which analysis and experiments on a specific technological process can be conducted.

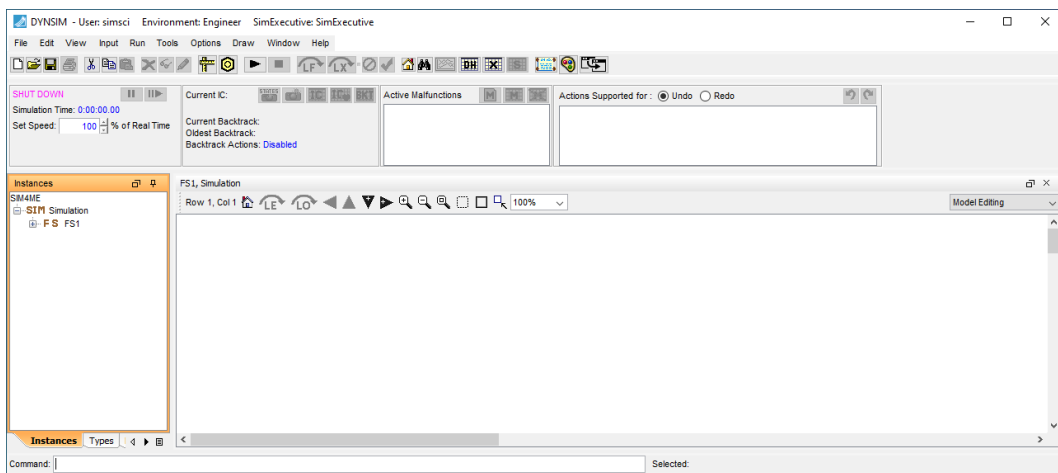


Figure 7 DYNsIM® environment

### 3.1.1. Units of Measurement

The initial step in setting up a new simulation in DYNsIM® is to select the units of measurement that will be used throughout the whole simulation file. The SI system is set as the default, and a custom name is assigned for a specific modeling project.

### 3.1.2. Components and Thermodynamics

At the start of the simulation, all chemical components involved in the process, including species and utility streams, must be defined. The flows used for equipment initialization must be taken into account to perform the most accurate simulation of a real plant. The source that will be used for the initialization of all process units consists of pure nitrogen in the gaseous state, due to its inert nature.

The DYNOSIM® software possesses a vast library of chemicals, and “ThermoLib\_4.3.271:SIMSCI” is used in this work, as it is the largest database of component properties, and the study focuses on specific compounds that are particularly characteristic of alcoholic beverages in the food industry.

Besides the chemical components themselves, the chemistry tab enables the user to select a thermodynamic model that describes the behavior of these substances throughout the simulated process. As mentioned in the previous section, the NRTL (Non-Random Two-Liquid) model is most suitable for representing mixtures similar to those considered in this study, so it was chosen in the Method section [12], [13].

### 3.1.3. Equipment

To design a flowsheet, the “Icon Pallet” is available in DYNOSIM®, which allows the user to select the desired plant element from a floating bar and place it in a specific position on the canvas. One can select not only principal units, such as distillation columns and pumps, but also more basic elements such as material and heat streams, valves and specific control instrumentation.

### 3.1.4. Instance Tree

On the left side of the canvas, there is the “Instance Tree”, a structured list that organizes all the components of the simulation. This file is subdivided into separate flowsheets, each containing the various models that are present in the canvas. By selecting a specific element in the Instance Tree, the user can access all its associated “point references,” such as process and design variables, organized in different categories. To monitor a particular variable during the simulation, it can be easily dragged and dropped onto the canvas, allowing its dynamic values to be displayed in real time.

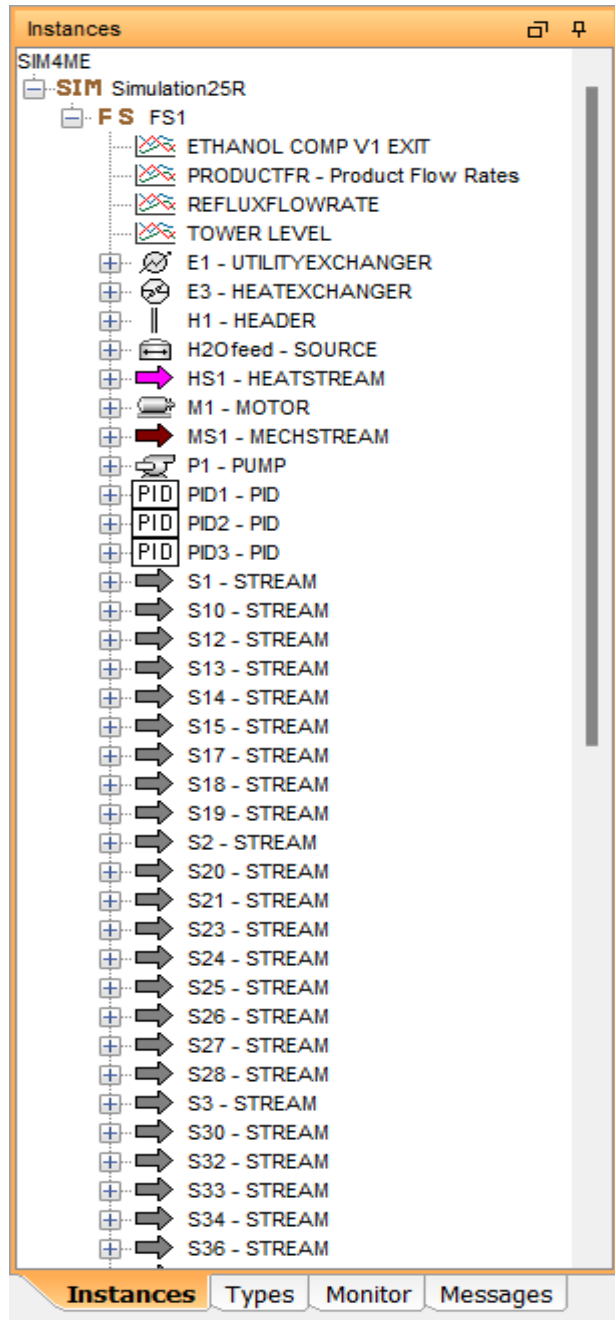


Figure 8 Instance Tree

### 3.1.5. DYN SIM® Toolbar

The engineering toolbar, located at the top left of the canvas, provides essential controls for managing the simulation. It allows the user to start, pause, stop, or accelerate the simulation up to 10,000 times the normal speed. Additionally, Initial Conditions (IC) can be created and saved,

enabling quick access at any point in the simulation, in order to save time on starting the process from the very beginning.

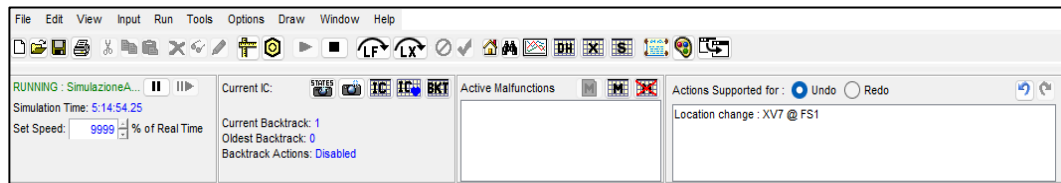


Figure 9 Main toolbar

## 3.2. Simulation

### 3.2.1. Scope of the study

As anticipated in the Introduction section, the objective of this study is to simulate the distillation of a water-ethanol mixture, representing the second distillation in the Cognac production process. The simulation reproduces the conventional batch distillation utilizing a Charentais alambic still. According to the classical technology, 2,500 liters of the mixture called *brouillis* are distilled, and the resulting product is divided into four separate fractions based on their alcohol content [26], [28].

The simulation is carried out in DYNOSIM®, replicating each phase of this process from start to finish.

Initially, the main objective is to develop a simulation that accurately reflects the actual process by creating reliable models for each unit and physical operation, ensuring that no numerical errors occur throughout the simulation. Once the scope of work is defined, the next step is to develop a detailed flowsheet of the process.

### 3.2.2. Flowsheeting

At this stage, the necessary nodes and connections are arranged on the canvas, and material and energy streams are added to construct the technological scheme.

The scheme developed in the previous year's thesis work is taken as a base and subsequently adjusted and expanded to align with the specific objectives of this study [34].

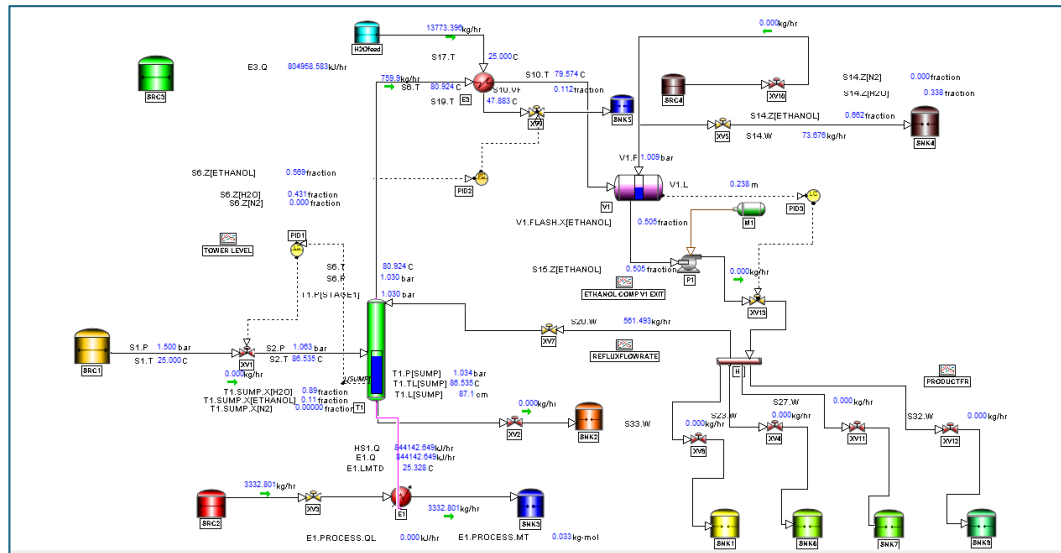


Figure 10 Flowsheet of the simulation process

In DYNsIM®, the simulation setup includes two key units that define the boundary limits of the process: the source and the sink, which, as the names imply, represent the beginning and the end of the material streams. Effectively, they perform the function of separating what happens inside and outside the process under study.

The batch distillation column is executed in a unit called Tower while Drum is used as the condenser vessel. To justify the choice of each component in the scheme, it is helpful to go through the entire technological process step by step.

### 3.2.2.1. Feed section

The key parameters of the distillate mixture, such as composition, pressure, and other essential data, are specified in the source unit (SRC1) through the Data Entry Window (DEW), presented in the Figure 11.

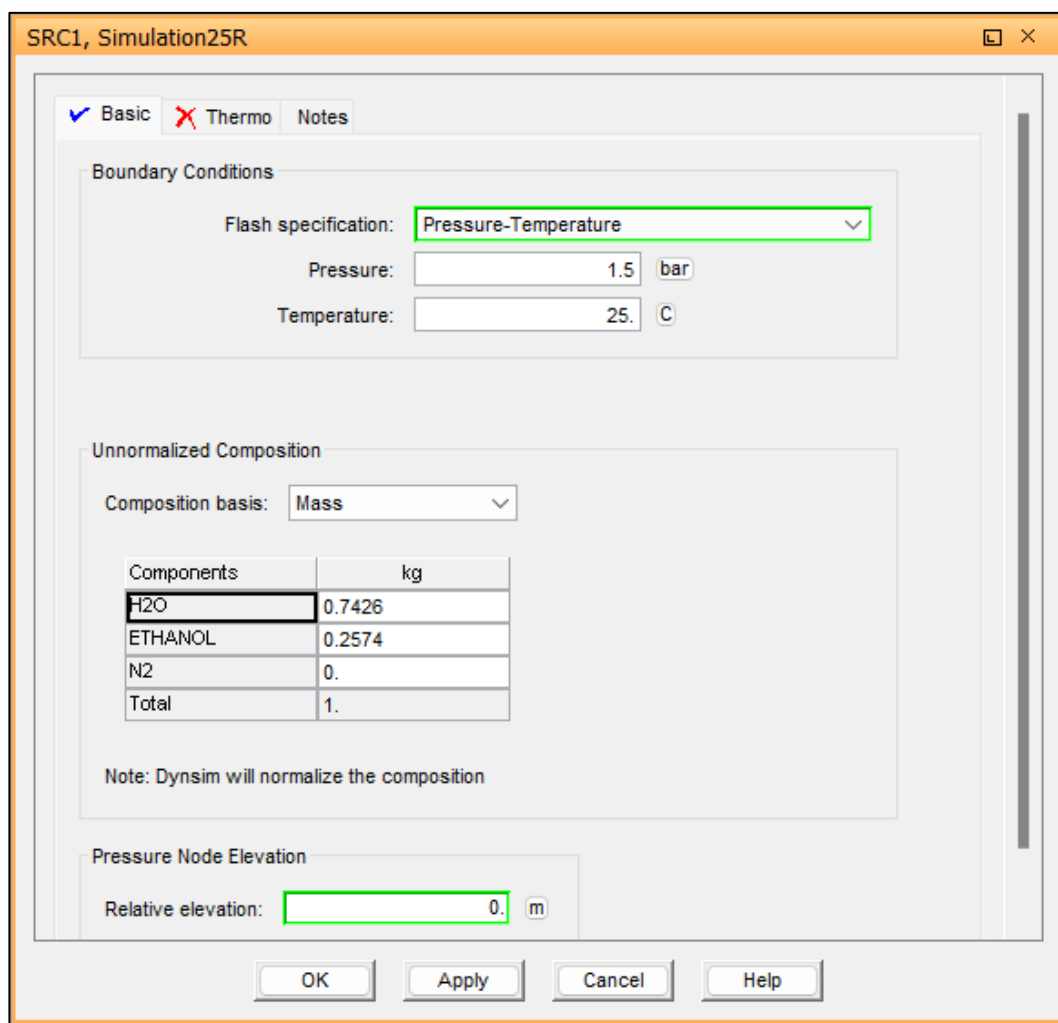


Figure 11 DEW with the key initial parameters

The appearing DEW displays different types of data to enter. Red windows are mandatory to input, green shows default values, and the orange ones are given by default but are recommended to check.

In the first case, the simulation is performed using a mixture of water and ethanol, representing *brouillis* – the intermediate fraction obtained during wine distillation in Cognac production. According to classical technology, its alcohol content ranges from 28 to 30% by volume. For simplification, a composition of 30% alcohol is selected.

In real conditions the batch of mixture is loaded into the apparatus at ambient temperature, and the entire process is carried out at atmospheric pressure, so the same conditions are maintained in the simulation.

A distinctive aspect of the simulator is that in order to guarantee a constant flow through valve XV1, a slightly increased pressure in SRC1 is set to ensure a positive pressure difference ( $\Delta P$ ).

Parameters	Values
Water, [ x mol (%vol)]	88,32 (70)
Ethanol, [x mol (%vol)]	11,68 (30)
Nitrogen [%mol]	0
Pressure [bar]	1.5
Temperature [°C]	25.0
Cv of XV1 valve	50

Table 1 Feed section parameters

As can be seen at the top right of the flowsheet (Figure 10), there is another source (SRC3) which the software automatically uses to initialize all the units correctly. At the start of the simulation, nitrogen gas is used to initialize the node units, conditionally filling all vessels and empty spaces with nitrogen at time  $t=0$ . To prevent system depressurization during shutdown, an additional nitrogen source (SRC4) is connected to the reflux drum.

Valve sizing is a crucial step, as incorrectly sized valves could potentially cause instabilities to the entire system. The target operating range for the valves is set between 15-80% of their maximum opening position. This range provides more precise control, reducing risks of cavitation and erosion while maintaining optimal flow regulation. The main parameter required for valve setup in DYNASIM®'s DEW is the valve sizing coefficient (Cv). The Reverse Flow Factor is set to 0 to prevent any reverse flow through the valve during the simulation.

Since the flowsheet is adapted from previous research, the Cv of each valve is manually adjusted to align with the specific requirements of the current simulation.

### 3.2.2.2. Distillation Column Section

To simulate a batch distillation process, the Tower unit is chosen in the DYNsIM® environment since it includes a sump, which acts as a working volume for the feed mixture undergoing the distillation process.

In order to simulate the heat flow from the reboiler an external heat stream input is connected to the sump. The Tower is designed to include plates, but since the goal is to simulate distillation in a Charentais alambic, only a single plate is specified. A bottom outflow stream with a valve is configured but remains inactive during the distillation process; it is only used to empty the column from the residues of the distillation.

Chemical reactions are not enabled in the simulation because analyzing them is beyond the scope of this particular work.

Next, the vapor and reflux streams are set up, each with its own valve, to connect the distillation column to the condenser section.

Parameter	Value
Number of trays	1
Tray spacing [m]	0.67
Sump spacing [m]	1.0
Column Diameter [m]	2.0
Initialization source	SRC3 (N <sub>2</sub> )

Table 2 Distillation column parameters

Below the column, the reboiler section is modeled using two vessels – a source and a sink – connected by a steam line that delivers heat to the liquid in the column’s sump through an energy stream E1.

The steam source (SRC2) provides low-pressure steam (LPS) at 1.5 bars and 130°C, while the sink (SNK3) is maintained at ambient pressure.

All the main parameters regarding the Reboiler section are summarized in the table below.

Parameters	Values
Pressure SRC2 [bar]	1.5
Temperature SRC2 [°C]	130
Cv of XV3 valve	400
Pressure SNK3 [bar]	1.0
Reboiler - Heat Exchanger E1	
Volume [m <sup>3</sup> ]	1
Heat Transfer Area [m <sup>2</sup> ]	50
Overall Heat Transfer Coefficient [kW/(m <sup>2</sup> K)]	0.2
Pressure node Elevation [m]	0
Initialization Source	SRC3 (N <sub>2</sub> )

Table 3 Reboiler section parameters

### 3.2.2.3. Condenser section

The condenser section is represented by a heat exchanger (E3) and a reflux drum. The vapors rising from the top of the column pass through the heat exchanger, where they are cooled by the cooling water and condensed into a horizontal vessel that also serves as the reflux drum. An additional cooling water line, consisting of a source (H2Ofeed) and a sink (SNK5), is added to regulate the coolant flow through the condenser via valve XV9. The reflux drum is configured to replicate the minimal reflux typically

observed during distillation in a Charentais Alambic, as outlined earlier in the Introduction. All the parameters discussed above about the Condenser Section are shown in the Table 4 below.

Parameters	Values
Condenser E3	
Heat Transfer Area [m <sup>2</sup> ]	50
Initialization Source	SRC3 (N <sub>2</sub> )
Overall Heat Transfer Coefficient [kW/(m <sup>2</sup> K)]	0.5
Pressure node Elevation [m]	2
Reflux Drum V1	
Diameter [m]	0.5
Height [m]	0.8
Thickness [mm]	12.7
Pressure node Elevation [m]	2
Initialization Source	SRC3 (N <sub>2</sub> )
Cooling Water Line	
H <sub>2</sub> Ofeed Pressure [bar]	3.0
H <sub>2</sub> Ofeed Temperature [°C]	25
Cv of XV9 valve	20
SNK5 Pressure [bar]	1.01

Table 4 Condenser section parameters

#### 3.2.2.4. Discharge section

The remaining liquid at the bottom of the column is discharged to the sink unit (SNK3). This stream is used either during ordinary plant shutdown after completing the distillation process or in the event of emergency shutdown.

On the top right of the flowsheet, a vent is placed to connect the top of the reflux drum (V1) to the ambient sink (SNK4) in order to release the incondensable gases, especially during the startup phase, when much nitrogen is present in the system.

#### 3.2.2.5. Product section

Finally, the last section in the process flowsheet is the one where the products of the distillation are collected. This section is composed of four sinks SNK1, SNK6, SNK7 and SNK8, each with a corresponding valve.

This section is linked to the rest of the plant via a line passing through the splitter H1 and valve XV13. The opening of each sink valve is adjusted based on the ethanol concentration in the reflux drum.

Parameter	Value
SNK2 Pressure [bar]	1.01
SNK2 Relative Elevation [m]	0
SNK1, SNK6, SNK7, SNK8 Pressure [bar]	1.01
SNK1, SNK6, SNK7, SNK8 Relative Elevation [m]	0
Cv of XV2 valve	100
Cv of XV8, XV4, XV11, XV12 valves	100

Table 5 Product and discharge sections

### 3.3. Control System

Batch distillation is generally more complex to control due to their dynamic behavior and numerous parameters involved compared to the continuously operating plants [9].

#### 3.3.1. Control Network

A control system is developed to obtain the required parameters and ensure the proper execution of the distillation process. To install the control, the PID is selected from the icon pallet, the control type (mass, level, temperature) is defined, and then it is connected to the flow, following the information path. The measurement instrument supplies the process variable to the controller, and the controller's output is directed to the valve.

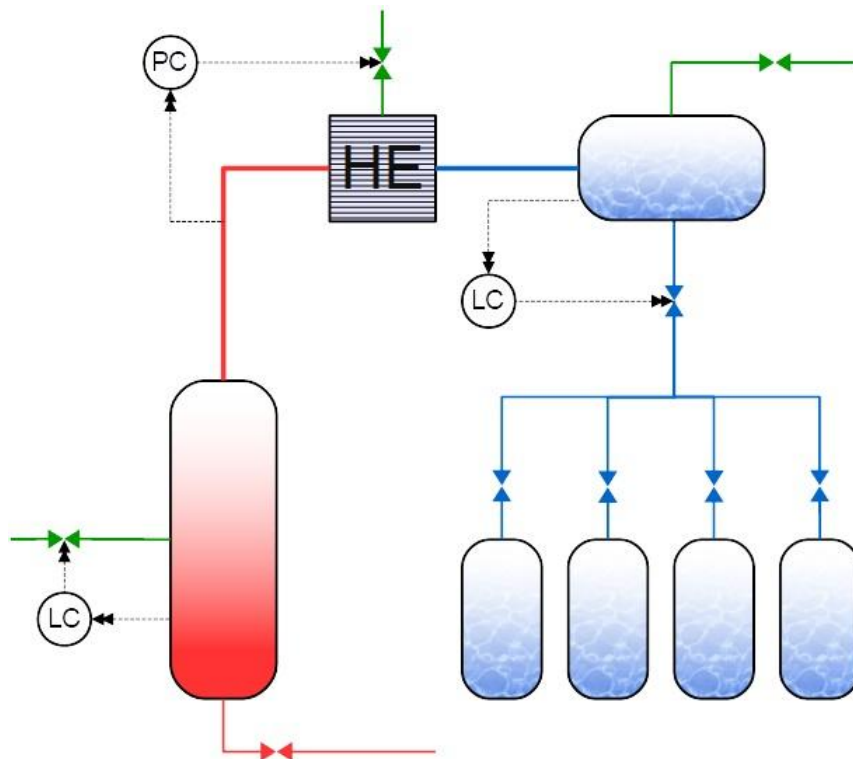


Figure 12 Control scheme

The first (PID1) is installed on a feed stream to control the filling of the Tower with a batch of distillation mixture. A level controller is set up in the sump to achieve the desired level based on the vessel's geometry. Once it is reached, the valve closes; the controller is switched to manual mode, and turned off as it is no longer needed during the rest of the process.

In the condenser section, two additional PID controllers are installed. PID2 is responsible for maintaining a steady pressure in the upper part of the Tower by adjusting the cooling water flow rate through Valve XV9. PID3 manages the Reflux drum level, acting on the valve XV13, ensuring it does not exceed the set point, which is defined as half of the horizontal vessel's volume.

No specific automated control is implemented to switch between distillate fractions during the simulation, as this reflects the traditional industrial approach used in real production. In practice, the distiller monitors the alcohol strength using an alcohol meter and relies on sensory evaluation – taste and aroma, to decide when to switch from one fraction to the next.

Below, in the Table 6, a summary of the controllers' variables, set point, action type, and parameters is provided.

	<b>PID1</b>	<b>PID2</b>	<b>PID3</b>
Controller type	Level	Pressure	Level
Controlled variable	Column sump level	Column top Pressure	Reflux drum level
Set point	0,88 m	1.02 bar	0.25 m
Manipulated variable	Feed flow valve	Cooling water valve	Product valve
Control action	Reverse	Direct	Direct
$K_p$	1	0.1	1

K <sub>i</sub>	0.005	0.005	0.005
----------------	-------	-------	-------

Table 6 Controller's summary

### 3.4. Scenarios

To avoid manual execution of the entire process, the Scenario function is implemented in the DYN SIM<sup>®</sup> menu. It provides instructions for the execution of all plant operations during certain time periods.

The conventional batch distillation process can be divided into four main stages: 1) Charging the mixture, 2) Heating and Reflux, 3) Production part, and 4) Shutdown and Discharge stage.

The time taken to complete all these processes, summed together, determines the total amount of time required to carry out the entire distillation.

#### 3.4.1. Charging the mixture

The first step is to load the cold feed mixture into the sump of the distillation column, in our case, the Tower.

Below are the code instructions provided to the software to perform the sequential actions needed to operate the plant. Some comments denoted by the “//” symbol are present throughout the code to provide a readable structure.

```
// Author: AnnaK
// Description: Filling of the sump
  a. // Initialization
SET XV5.OP = 0.8;
SET XV3.OP = 0;
SET XV9.OP = 0;
```

```
SET XV10.OP =0;
SET XV13.OP = 0;
SET XV7.OP = 0;
SET XV8.OP = 0;
SET XV4.OP = 0;
SET XV11.OP = 0;
SET XV12.OP = 0;
SET PID1.SDCS=0;
SET PID1.MA=0;
SET PID3.SDCS=0;
SET PID2.SDCS=0;
SET PID2.MA=1;
SET PID3.MA=1;
SET PID1.MANSET=0.8;
    b. // Loading
RUN;
WAIT 5;
SET SETSPD=9999;
WAIT UNTIL T1.VOLL[SUMP]>2.5;
SET SETSPD=100;
//Activate PID1 on Manual Close
SET PID1.MANSET=0;
SET XV1.OP = 0;
FREEZE;
PROMPT "The Sump is filled and feed valve close. Ready for heating";
```

Before starting the simulation, all units are set to their initial states. By default, controllers 2 and 3 are set to AUTO mode in DYNOSIM®, while PID1 is switched to manual mode to maintain a constant flow until the liquid level in the sump reaches the set point.

All valves are closed, except for two: XV1, which remains open to allow the mixture to enter the column, and XV5, the vent, which lets nitrogen exit the system as the sump fills. This is done using the command “.OP=0,” where 0 and 1 represent fully closed and fully open positions, respectively, and any value in between indicates a partial valve opening.

Once all controllers and valves are configured, the simulation is initiated using the “RUN” command. DYNOSIM® calculates the numerical solutions for the differential and algebraic equations of the models, updating the values of each variable every 0.25 seconds by default. To accelerate the process, the simulation speed is increased with the command “SET SETSPD=9999;”.

As soon as the simulation is started, the valve XV1 is opened, providing the feed into the column, controlled by the PID1, the level starts to increase. The command “WAIT UNTIL T1.VOLL[SUMP]>2.5” allows for detection when 2.5 m<sup>3</sup> of feed mixture are loaded into the column’s sump, and as soon as this condition is satisfied, the next command can be executed. When the desired load is reached, PID1 is turned off, and the valve XV1 is manually set to a completely closed position. After completing the first scenario, the new set of instructions can be executed.

### 3.4.2. Heating and Reflux

The second scenario manages the crucial startup phase, namely the heating of the mixture in the Tower sump.

```
// Author: AnnaK
```

```
// Description: Heating and Reflux
```

```
a. // Initiate heating
```

```

SET XV3.OP = 0.5;

RUN;

SET SETSPD=9999;

WAIT 1210;

FREEZE;

PROMPT "Bubble T reached. Now Fill the Condenser Vessel";

    b. // Fill the Reflux Drum

RUN;

SET SETSPD=1000;

WAIT UNTIL V1.L>0.25;

FREEZE;

    c. //Reflux

PROMPT "Start the Reflux";

SET M1.CL=1;

SET M1.OP=0;

SET SETSPD=100;

SET XV7.OP=0.8;

RUN;

SET SETSPD=1000;

WAIT UNTIL V1.FLASH.X[ETHANOL]>0.505;

FREEZE;

SET XV7.OP=0;

PROMPT "The Reflux is finished and Reflux valve close. Ready for
Production";

```

The valve XV3 supplying the heat from the steam line is open for 50%, and some time later, the boiling temperature, which is an important parameter in the heating process, is reached [35].

The vapor rises from the top of the distillation column, it passes through Heat Exchanger 3, where it is cooled by cold water and condenses, filling the reflux drum. Once the level in the reflux drum reaches the specified set point, which is realized by the command "WAIT UNTIL V1.L > 0.25;", the reflux stage begins. Its objective is to increase the ethanol concentration in the vapors to the value required to start the first distillate collection step. The reflux valve XV7 is set to 80%, the pump is activated using the commands "SET M1.CL=1;", "SET M1.OP=0;". Once the condition is met, with a command "WAIT UNTIL V1.FLASH.X[ETHANOL]>0.505;", the reflux stage is finished, the reflux valve is closed, and the system is ready for the production stage.

### 3.4.3. Production Part

The following scenario executes the next series of operations in the process.

```
// Author: AnnaK
```

```
// Description: Production Part
```

```
  a. //Heads collecting
```

```
SET XV3.OP = 0.3;
```

```
SET XV8.OP = 0.5;
```

```
RUN;
```

```
SET SETSPD=9999;
```

```
WAIT UNTIL V1.FLASH.X[ETHANOL]<0.48;
```

```
FREEZE;
```

```
PROMPT "Heads are collected";
```

b. //Hearts collecting

SET XV8.OP = 0;

SET XV4.OP = 0.5;

SET XV3.OP = 0.07;

RUN;

SET SETSPD=9999;

WAIT UNTIL V1.FLASH.X[ETHANOL]<0.316;

FREEZE;

PROMPT "Hearts are collected";

c. //Seconds collecting

SET XV4.OP = 0;

SET XV11.OP = 0.5;

SET XV3.OP = 0.7;

RUN;

SET SETSPD=9999;

WAIT UNTIL V1.FLASH.X[ETHANOL]<0.016;

FREEZE;

PROMPT "Seconds are collected";

d. //Tails collecting

SET XV11.OP = 0;

SET XV12.OP = 0.5;

SET XV3.OP = 0.5;

RUN;

SET SETSPD=9999;

WAIT UNTIL V1.FLASH.X[ETHANOL]<0.009;

FREEZE;

```
PROMPT "Tails are collected";  
RUN;  
SET XV12.OP = 0;  
SET M1.CL=1;  
SET M1.OP=0;  
SET SETSPD=100;  
SET XV7.OP=0.8;  
FREEZE;
```

The production phase continues as heat is supplied through the opening of valve XV3, maintaining the distillation process. This phase takes a lot of time, so the simulation speed can be increased. The transition between different distillate fractions is controlled by the command "WAIT UNTIL V1.FLASH.X[ETHANOL]<...;" based on the ethanol content in vessel V1.

After the last fraction is collected, the system prepares for the shutdown phase.

#### 3.4.4. Shutdown and discharge

Finally, the last set of instructions is shown in order to empty the reflux drum and the column from the residual liquid.

```
// Author: AnnaK  
// Description: Shutdown and Discharge stage  
  a. //turn off steam flow  
RUN;  
SET XV3.OP=0;  
//PID3 deactivate  
SET PID3.SDCS=0;
```

```

SET PID3.MA=0;
    b. //close CW
WAIT 10;
FREEZE;
SET PID2.SDCS=1;
SET PID2.MA=0;
SET PID2.MANSET=0;
SET XV9.OP=0;
PROMPT "CW off";
    c. //close the vent
SET XV5.OP=0;
    d. //empty the reflux drum
SET PID3.SDCS=1;
SET XV7.OP=0.9;
    e. //nitrogen open
SET XV10.OP=0.8;
RUN;
WAIT 5;
PROMPT "Nitrogen open";
SET SETSPD=9999;
WAIT UNTIL V1.L<0.02;
    f. //shut down the motor that feeds the pump
SET M1.CL=0;
SET M1.OP=1;
SET SETSPD=100;
FREEZE;

```

```

PROMPT "Pump off";
RUN;
    g. //discharge the column
SET XV2.OP=0.8;
WAIT 10;
SET SETSPD=9999;
WAIT UNTIL T1.L[SUMP]<0.009;
SET SETSPD=100;
SET XV10.OP=0;
SET XV7.OP=0;
SET XV2.OP=0;
WAIT 3;
FREEZE;

```

First, the heat supply is stopped by closing the steam valve XV3. Once heating and evaporation stop, the cooling water flow is also shut off. The level in the reflux vessel no longer raises, the PHD2 and PID3 controllers are no longer required, and are switched to the SDC OVERRIDE mode. The vent is closed by the "SET XV5.OP=0;" command. At the same time, another line supplying nitrogen to the system is opened via XV5, allowing to fill the reservoirs during the discharging phase and maintain the constant pressure in the system. The command "WAIT UNTIL V1.L<0.02;" empties the reflux drum, after that, the motor of the pump is turned off. The Tower sump is discharged by opening the valve XV2 and using the "WAIT UNTIL T1.L[SUMP] < 0.009;" command. Finally, the scenario freezes the simulation, marking the end of the process.

# 4 Results and Discussion

In this chapter, the results of dynamic modeling in a batch distillation system are presented.

The data obtained using the Trends tool in DYNOSIM®, which allows exporting time series data in the .csv format and conducting further analysis in Microsoft Excel is utilized to build the graphs. As mentioned in previous chapters, DYNOSIM® tracks and calculates a wide range of process variables, including component compositions, molar flow rates, pressures, temperatures, and liquid holdups. These parameters provide a comprehensive view of the system's behavior over time and are crucial for understanding the dynamics of the process.

However, more specific variables are monitored by using special functions in order to provide the desired trends as output.

## 4.1. Dynamic Simulation of the Second Distillation

### 4.1.1. Initial Process Dynamics

The first stage begins with filling the sump of the distillation column with a volume of 2500 liters, which corresponds to the typical batch size used in Charentais distillation. In France, producers often limit the second distillation to 2500 L batches, as this facilitates better control over the separation of fractions.

The mixture used in the distillation represents *brouillis*, the intermediate fraction composed primarily of water and ethanol, obtained during the wine distillation stage in Cognac production. According to classical technology, the alcohol content of *brouillis* is around 28%. However, since it is often distilled together with the *secondes*, its ethanol concentration

may rise to approximately 28.5–30% by volume. In this simulation, a composition of 30% alcohol has been selected [36] [26], [28], [29].

This volume corresponds to a liquid level of 0.88 meters in the sump, calculated based on the diameter and height of the distillation column. Figure 13 (Tower Level) illustrates the dynamics of the sump filling process. Once the target level is reached, valve 1 closes. The PID1 controller is deactivated and is no longer used for the rest of the process cycle.

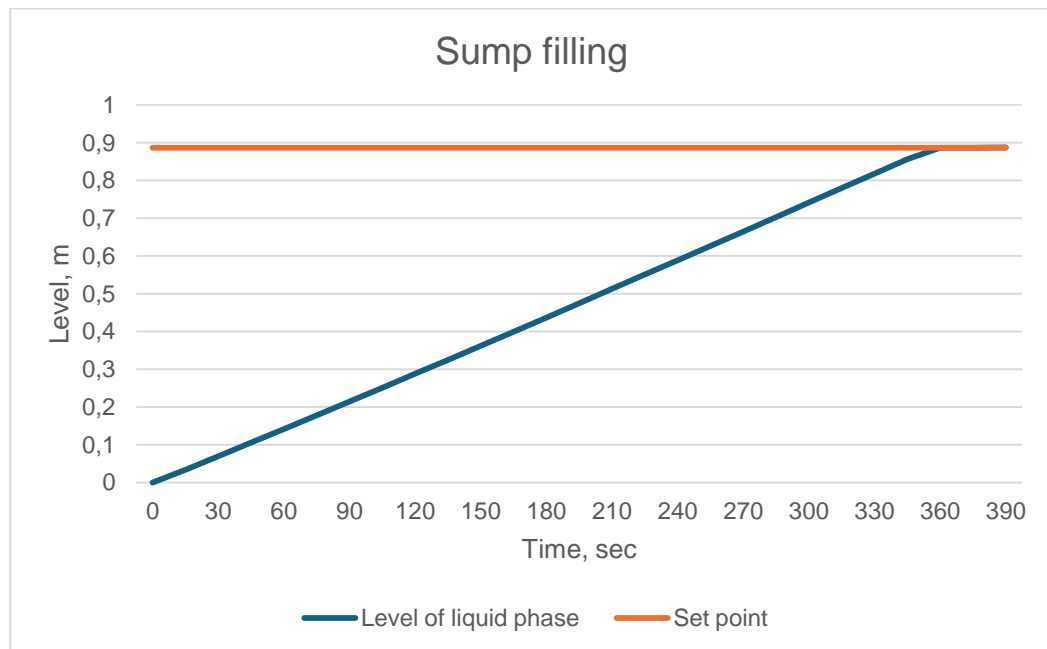


Figure 13 Sump filling

According to the Figure 13, the filling process takes approximately 6 minutes in the simulator to complete the initial step. Since the process vessels are initially filled with nitrogen, the first action required for proper filling is to open the vent valve XV5, allowing the gas to be released.

#### 4.1.2. Heating Followed by Reflux

Heating begins with the partial opening of valve 3 (at 50%), initiating the transfer of heat to the sump from the heating section. As a result, the temperature gradually rises until it reaches the boiling point.

The mixture can be roughly described as a binary solution consisting of water and ethanol. The boiling points of the pure components are 100°C for water and 78.5°C for ethanol. This is a homogeneous mixture, as it forms a single liquid phase. According to literature sources, the boiling point of the specific water-ethanol mixture considered in this work is around 86°C under atmospheric pressure [12].

The heating process continues while simultaneously controlling the pressure at the top of the column to ensure it does not exceed the critical value. At the same time, the flow of cooling water is regulated by adjusting valve XV9 through the PID2 controller.

The liquid condenses after the heat exchanger 3 and is collected in the vessel 1 (Reflux drum).

The next milestone in the process is to reach the set point level of the condensing distillate in the drum, which is maintained at 0.25 m. The third controller, PID3, is responsible for achieving this goal. Once the set level is reached, the reflux process is initiated.

At first, there were some doubts about including the reflux process in the simulation. However, a closer look at the classical Charentaise distillation technology reveals that a brief reflux is indeed present, due to the design of the distillate apparatus – the traditional alambic still. As mentioned in the Introduction, the part called the *chapeau*, located just below the heating section, has a special shape. This design causes some vapors to condense on its large surface and partially return to the boiling liquid before leaving the heating zone. This allows for an increase in the concentration of the volatile components through vapor-liquid exchange, similar to what happens in a real distillation column.

The Charentais alambic does not use plates, and to keep the simulation as close to the real process as possible, no plates were added to the column in the model either. Instead, the implemented reflux imitation is the only way to realistically reproduce the separation conditions characteristic for this type of distillation. This approach is also essential from a technological point of view: the ethanol concentration in the distillate collected in the reflux drum is initially not high enough to start the main

production phase. According to classical technology, this phase should only start once the ethanol content reaches 78%.

To initiate the reflux process, valve 7 is opened. Since the pressure in both the drum and the column is roughly equal, a pump is used to create the necessary flow back into the column. This is achieved by applying mechanical force to the pump via the mechanical flow MS1.

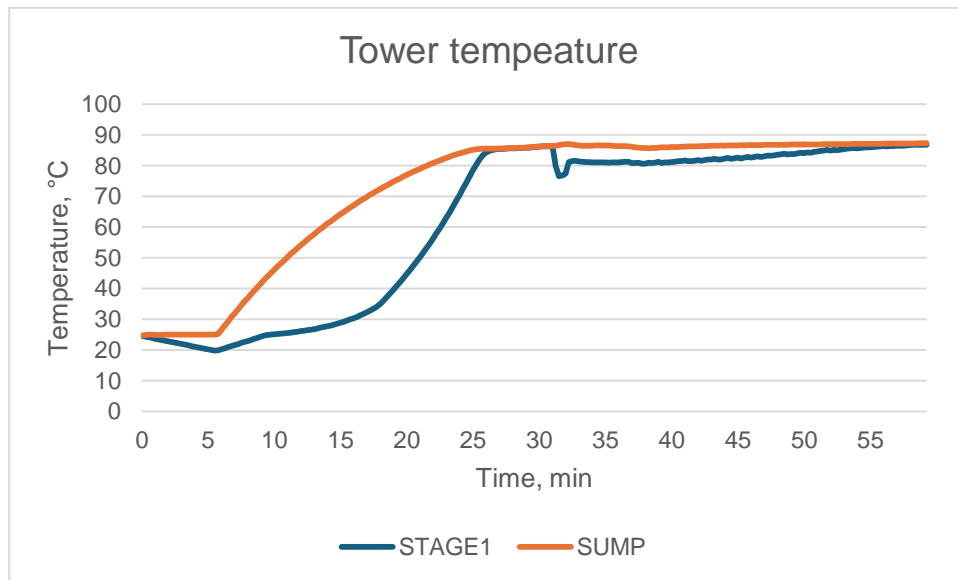


Figure 14 Tower sump and stage temperature

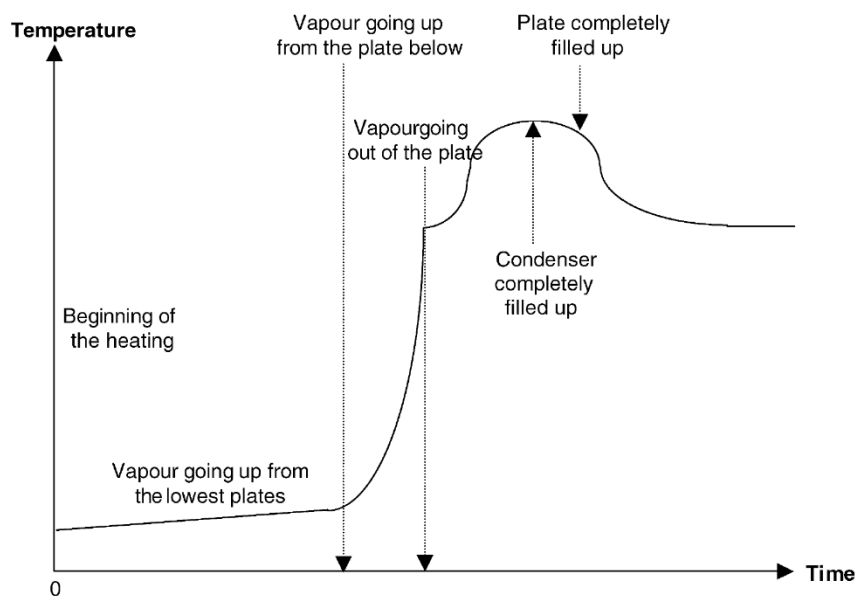


Figure 15 Tray temperature dynamics [9]

The Figure 15 illustrates the heat transfer dynamics within the distillation column, where the sump is connected to the heat source and heat is carried upward by the rising vapor. The temperature profile clearly reflects the point at which the reflux process begins, as the cooled liquid starts returning to the top of the column. The result generated by the dynamic simulator follows the same trend as observed in Figure 14.

The short-term reflux continues until the ethanol concentration in the liquid reaches the value of 0.505 molar concentration, which corresponds to 78% by volume. Once this threshold is achieved, the valve closes, the reflux process ends, and the simulation system becomes ready for the main production phase — the collection of distillate fractions based on ethanol concentration.

#### 4.1.3. Production Part

As briefly mentioned earlier, Cognac distillation is carried out in two stages. The first stage, known as wine distillation (WD), involves distilling the wine to produce an intermediate mixture called *brouillis* or "low wine." Once a sufficient volume of *brouillis* is collected, it undergoes a second distillation (BD), known as "*bonne chauffe*", to produce the final product. This process is commonly known as double distillation.

This study focuses on the second distillation stage, during which the distillate is divided into four main fractions: head, heart 1 (which is the Cognac itself and will later be aged in barrels), heart 2, or *secondes*, and tail. The alcohol concentration in the distillate is highest in the first fraction — around 80% by volume — and gradually decreases, reaching 0% by the end of the process, as presented in Figure 6.

In this simulator, the fraction collecting process is modeled by continuously measuring the ethanol molar concentration in the reflux drum. Based on this value, the system switches between the sinks for collecting the final product, opening and closing the corresponding valves. During this phase, the distiller plays a key role, monitoring alcohol concentrations and assessing the quality during the process [21].

At the beginning of the production stage, valve XV8 opens to allow the collecting of the head fraction.

The first cut is made when the ethanol content in the distillate reaches 75% by volume, which corresponds to a molar concentration of 0.480. This threshold is integrated into the production stage scenario. Once this value is reached, the command closes valve XV8 and opens valve XV4, initiating the collecting of the second heart fraction – the Cognac itself. This fraction continues to be collected until the ethanol concentration drops to 0.316, which corresponds to 60% by volume.

This is the longest stage of the distillation process, typically lasting 6 to 7 hours. In a traditional Charentais alambic, it yields a product with an average alcohol content of around 70% by volume.

The simulator signals the completion of the heart collection, at which point valve XV4 closes and valve XV11 opens. The next cut is made when the ethanol molar concentration drops to 0.016, corresponding to 5% alcohol by volume. At this point, the *secondes* are collected in sink SNK7. Valve XV11 closes and valve XV12 opens, allowing the collecting of the final fraction – the tails. The process continues until all remaining alcohol has been distilled. The production stage is considered complete when the ethanol molar concentration in vessel 1 falls to 0.009.

By adjusting the heating parameters in the DYNOSIM® simulation, it is possible to reproduce process durations that closely match those observed in real-life alambic distillation. A comparison of the time parameters from the classical Charentais method and the DT simulation is presented in Table 7.

<b>Duration of stages of production process</b>	<b>Classical technology</b>	<b>Digital Twin</b>
Collecting of heads	30 min	23 min
Collecting of hearts	6 h	6 h 02 min
Collecting of <i>secondes</i>	4 h 30 min	4 h 45 min

Collecting of tails	1 h	1 h 03min
---------------------	-----	-----------

Table 7 Time parameters of the real process and the DT simulation

In total, the second distillation may last more than 12 hours.

As the ethanol concentration in the boiling liquid decreases, its boiling point increases, as can be seen in the Figure 16.

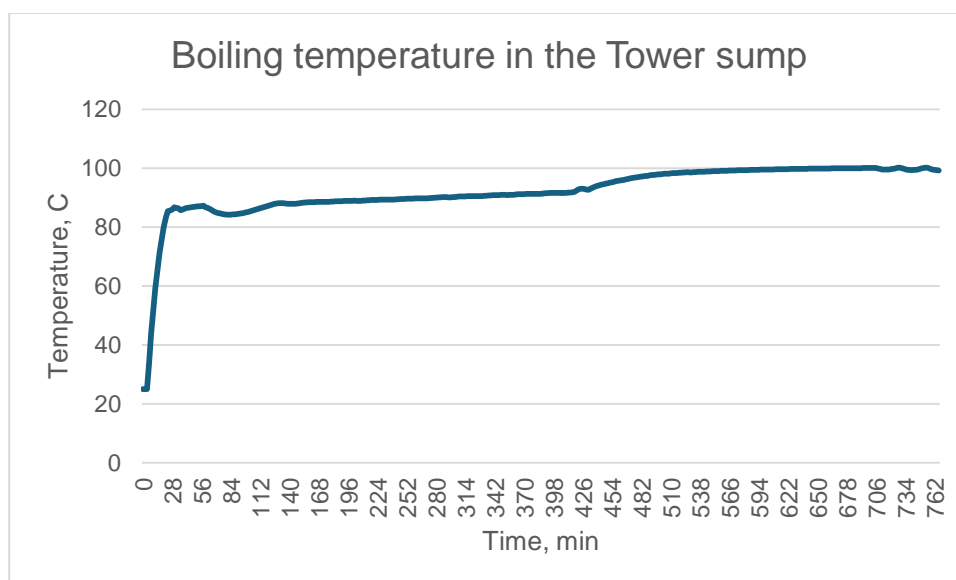


Figure 16 Tower sump temperature during the simulation

#### 4.1.4. Shutdown and Discharging Part

Once all product fractions have been collected, the shutdown phase begins. The first step is to stop the heating by closing the steam valve XV1. As a result, no more vapors are generated. After a short delay, the cooling process is also interrupted by closing valve XV9, which cuts off the flow of cooling water.

The liquid remaining in the column no longer contains ethanol but is rich in heavier volatile compounds that are undesirable in the final distillate. Therefore, it must be discharged, along with other residual contents in the reflux drum. To do this, the contents of the reflux drum are first returned to the column via the reflux line. Once combined, the entire volume,

including the residue from distillation, can be removed. However, before opening the discharge valves, it is necessary to supply nitrogen to the system through valve XV10 from source SNK4. This step is essential to avoid the pressure issues of the system, and ensure safe discharging.

Once the pump transfers all the remaining liquid from the reflux drum back into the column, it is switched off. Then, by opening valve XV2, the contents of the column are discharged. At this point, the system is fully emptied and ready for a new batch of feedstock.

## 4.2. Behavior of volatile Compounds in Simulation

The behavior of volatile compounds during the batch distillation of wine or *brouillis* in traditional Charentais copper stills has been the subject of numerous studies, using a variety of physical and analytical methods [18], [22], [24], [28], [37], [38], [39], [40].

As noted above, one of the main ways to separate compounds during batch distillation is to split the distillate into fractions collected over time.

The head fraction is rich in highly volatile compounds, including both undesirable compounds and those of interest. As many undesirable compounds give the distillate an unpleasant, strong, and pungent flavor, this fraction must be separated.

The heart is the most desirable part of the distillate; it is rich in ethanol and carries a pleasant aroma. After aging, this is the part that becomes the final spirit. The tail, collected at the end, contains heavier, oily compounds that are generally undesirable. The head and tail fractions are usually wholly or partially recycled in the following run to optimize ethanol recovery. In practice, the decision on when to make each cut is typically based on ethanol concentration or sensory evaluation – an approach which depends on the distiller's experience. However, this empirical method alone is insufficient without a solid understanding of the different behavior of the volatile compounds during distillation and the influence of process parameters [41].

In this work, with the help of DYNOSIM® software, a simulation is produced, examining the behavior of the aroma compounds during the distillation. The focus is on tracking their distribution across the four distillate fractions, based on how their concentration profiles evolve over time.

The simulation results are subsequently compared with those reported in previous studies to assess how accurately the software can reproduce the real behavior of volatile compounds during distillation. When performing these future steps, it is important to understand, if the simulation program can capture the changes in the mixture composition, given that the congeners are often present in tiny amounts.

At the start of the simulation, it is noticed that the boiling point dropped by nearly 1 degree, from 86°C to around 85°C. This may likely indicate that volatile compounds with lower boiling temperatures are added.

From the wide range of substances present in alcoholic beverages at varying concentrations, 5 volatile compounds: acetaldehyde, ethyl acetate, methanol, isoamyl alcohol and furfural, are selected for this study. These particular compounds are chosen primarily because their relatively high concentrations increase the probability of accurately tracking their behavior through simulation. Additionally, they represent diverse chemical families, providing a broader understanding of the distillation process. Each volatile component distills based on to the following criteria: boiling point, relationship with alcohol or water, and the variation of alcohol content in the vapor during the distillation.

According to the concentration profiles as a function of decreasing ethanol concentration (in % v/v) in the distillate for the wine and *brouillis* distillations, the substances are conditionally divided into types (see Figure 17) [29], [35] [28].

It is remarkable that the classification differs for wine (WD) and low wine (BD) distillation (Table 8).

Classification of compounds during low wine distillation	
1	Acetaldehyde, 1,1-diethoxy-ethane, 1,1-diethoxy-methyl-2-propane, ethyl acetate, ethyl propanoate, ethyl butanoate, hexyl acetate, ethyl caproate, ethyl laurate, ethyl myristate, ethyl palmitate, isobutyl caprate, isoamyl acetate, isoamyl caprylate, isoamyl caprate, isoamyle myristate
2	2-Phenylethanol
3	Methanol
6	Furfural, 2-phenylethyl acetate, ethyl lactate, diethyl succinate, caprylic acid, capric acid, lauric acid
7	1-Propanol, 2-methyl-1-propanol, 1-butanol, 2-methyl-1-butanol, 3-methyl-1-butanol, ethyl stearate, ethyl oleate, ethyl linoleate, ethyl linolenate
8	Ethyl caprylate, ethyl caprate.

Table 8 Classification of compounds during low wine distillation

Type 1 components, which distill first, have low boiling points and are soluble in alcohol. Examples include acetaldehyde, with a boiling point of 21°C, and ethyl acetate, which boils at 77°C. Most of these compounds evaporate at the start of the distillation process, resulting in high concentrations in the heads and at the beginning of the heart fraction.

Type 3 components, found both in the heads and the heart fractions, have relatively low boiling points and are soluble in alcohol, with varying solubility in water. Methanol, which boils at 65.5°C, is an example of a Type 3 compound.

Type 6 components, which also appear during distillation, have high boiling points and are highly soluble in water. They begin to distill in the middle of the heart fraction. Furfural, with a boiling point of 161°C, is an example of this type. Its concentration gradually increases from the middle of the heart through to the tails.

Some higher alcohols classified as Type 7 exhibit behavior similar to Type 3 components, representing relatively volatile compounds that concentrate

in the early fractions of the distillate. A typical example is 3-methyl-1-butanol, or isoamyl alcohol, which has a boiling point of 132°C [28], [35].

This classification helps to explain the general behavior of different compounds throughout the distillation process, as illustrated in the Figure 17.

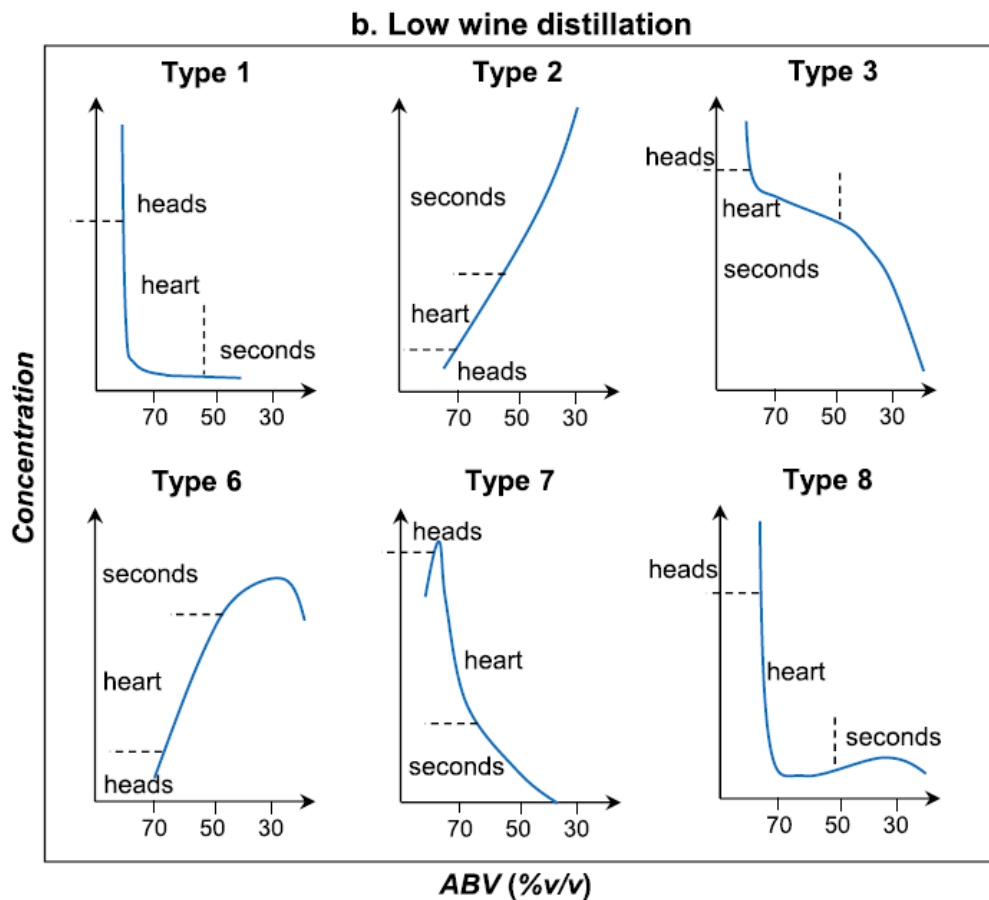


Figure 17 Concentration profiles of volatile compounds during the low wine distillation

The main challenge was to determine the appropriate amounts of components for testing in the simulation program. On the one hand, the concentrations had to be high enough for the program to produce visible results. On the other hand, it was important to maintain realistic concentration levels to reflect actual conditions. Literature data vary widely in their reported values, and while abundant information exists on

raw wine and the final product, there is very little data available on the composition of the intermediate product, *brouillis* [20], [35], [37], [42], [43] [29], [30].

Therefore, the final composition of the low wine for simulation analysis was determined carefully and almost empirically. It is presented in the Table 9.

<b>Component</b>	<b>Value, mg/L</b>
Acetaldehyde	1000
Ethyl Acetate	1000
Methanol	1500
Isoamyl Alcohol(3-methyl-1-butanol)	500
Furfural	100

Table 9 Composition of the mixture for simulation

Initially, the idea was to examine all the previously mentioned components within the same simulation. However, since their concentrations differ by an order of magnitude, it was hardly possible to clearly observe the behavior of each individual substance in the mixture in a representative way.

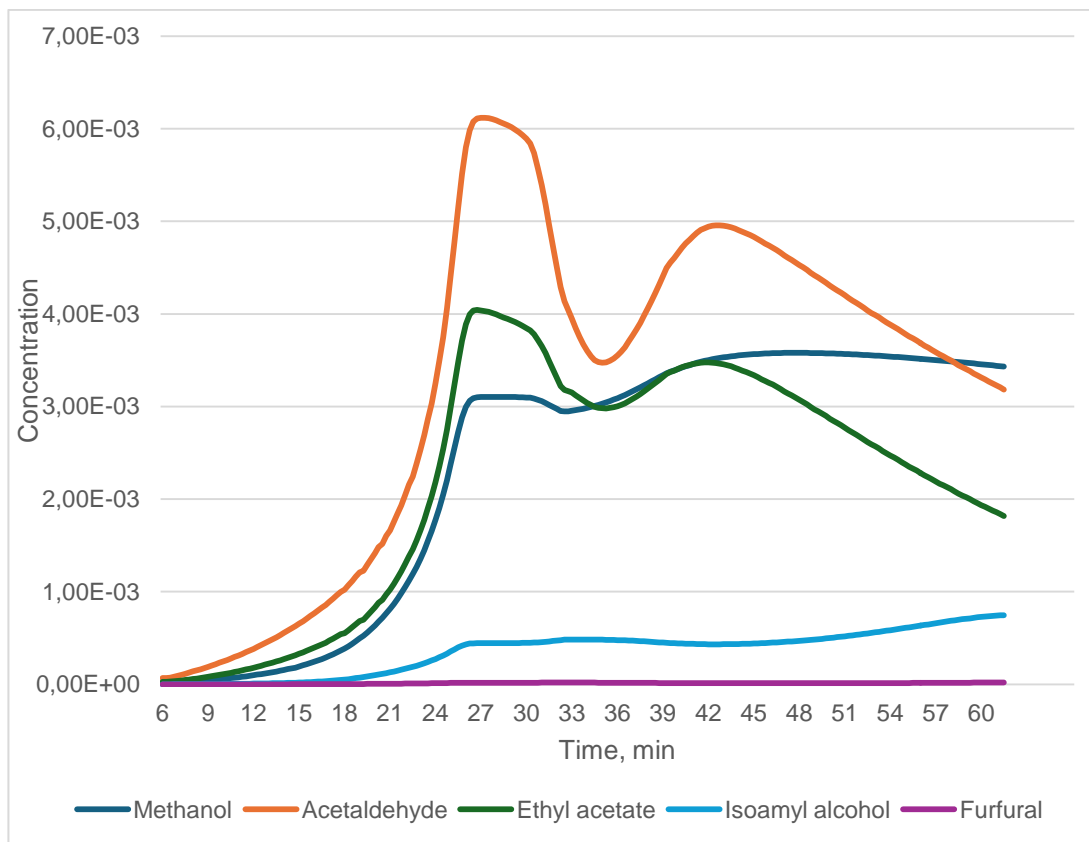


Figure 18 Volatile compounds behavior during the initial stages of the distillation process: heating and reflux

Nevertheless, the combined graph shows how the selected components behave during the initial stages of the distillation process: heating and reflux. In the simulation, as the temperature rises, the concentration of volatile compounds in the vapor begins to increase, just as it does in a real distillation process. This concentration peaks around the boiling point. One particularly interesting aspect to observe is how these volatiles behave during the reflux stage. Compounds with the lowest boiling points exhibit the highest volatility, and the reflux process helps to increase their concentration by returning part of the condensed vapor back into the column. Since these compounds are removed during the collection of the head fraction, this step plays a crucial role in eliminating substances with unpleasant odors, ultimately improving the quality of the final spirit. Due to both its low concentration and its specific behavior, furfural is not very clearly represented on the combined graph. However, as will be shown

later, its concentration gradually increases during the distillation process. The next step is to run separate simulations for each component individually in order to better observe and analyze the specific behavior patterns characteristic of each substance.

#### 4.2.1. Acetaldehyde

As mentioned earlier, acetaldehyde belongs to Type 1 compounds. In the simulation, it clearly demonstrates its typical behavior: a high concentration right at the start of the distillation, followed by a sharp decline as the process continues.

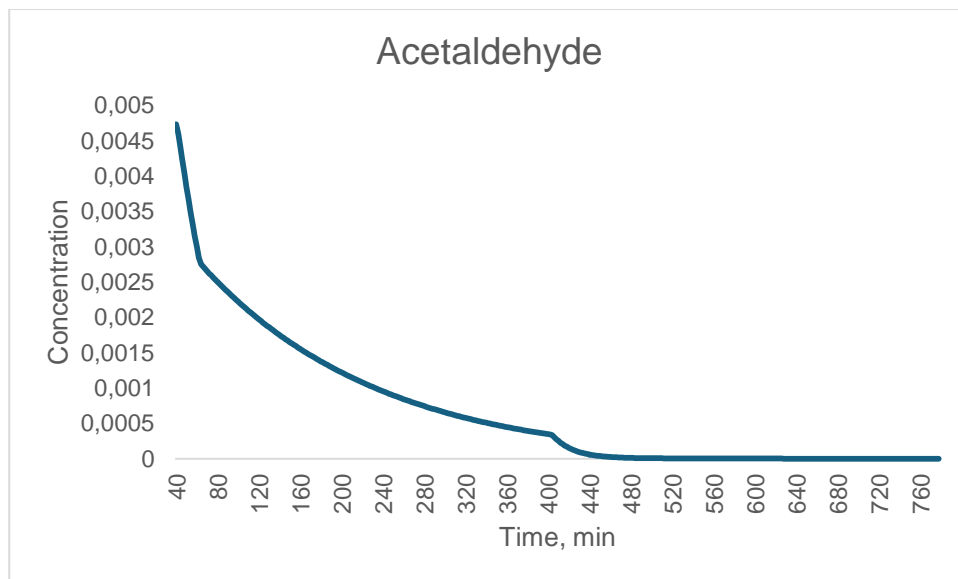


Figure 19 Acetaldehyde concentration dynamics

#### 4.2.2. Ethyl acetate

Ethyl acetate belongs to the same Type 1 as acetaldehyde, and like most esters, has a higher volatility than ethanol, so it is mostly concentrated in the head fraction. In large quantities, it can produce a sharp, glue-like aroma, whereas in smaller concentrations, it gives a pleasant flavor to the distillate.

Because of this it is critical to determine the precise moment to separate the head from the heart fraction. In the simulation, ethyl acetate shows a

similar pattern — its concentration peaks at the beginning and then steeply drops as the distillation goes on.

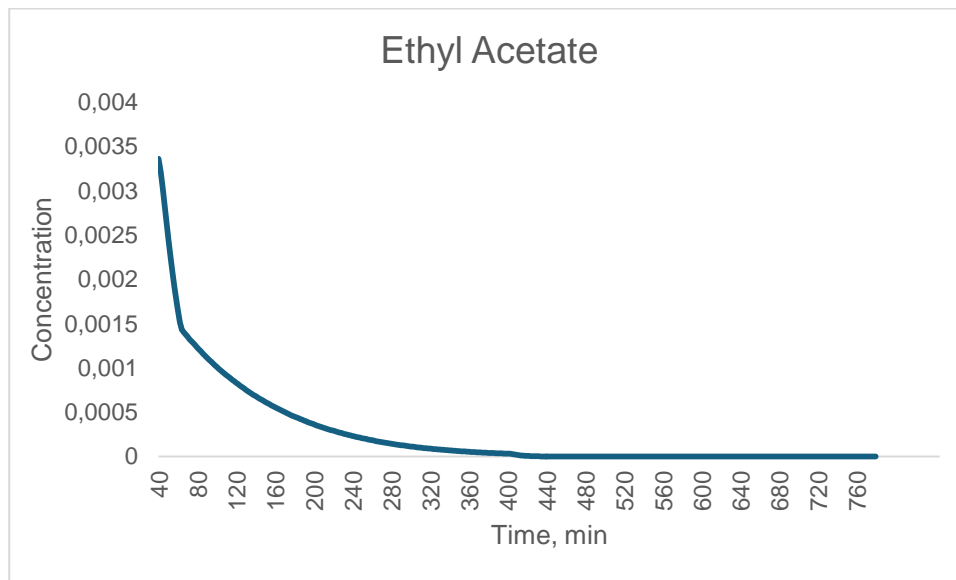


Figure 20 Ethyl acetate concentration dynamics

#### 4.2.3. Methanol

Given methanol's low boiling point ( $64.7^{\circ}\text{C}$ ) and high volatility, it would be reasonable to expect that its highest concentrations occur in the head fractions, distilling mainly at the start of the process and then steadily decreasing as distillation progresses. However, according to literature data [44] methanol concentrations can show an unexpected increase toward the end of the distillation.

Investigating the methanol's behavior during distillation, it can be noted that separating it from ethanol is rather hard because their volatilities are very similar. This is likely due to the association phenomenon, where hydrogen bonds are created between water molecules and alcohols, affecting their evaporation patterns, which was confirmed by other authors [33].

As a result, the hearts contain this undesirable compound, which is subject to restrictions due to its high toxicity and strict regulations limiting its presence in alcoholic beverages.

The simulation in the DYNOSIM® program confirms that methanol is present in all fractions obtained during distillation.

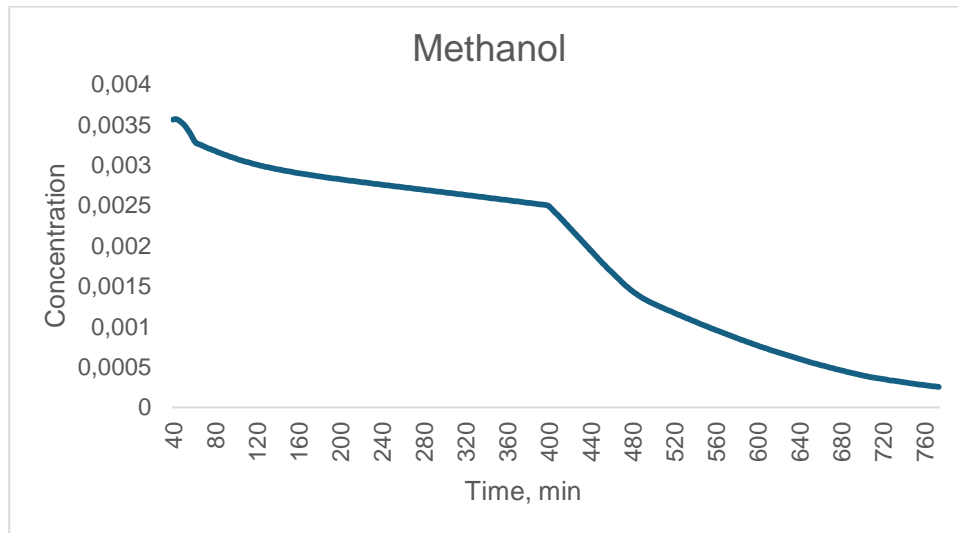


Figure 21 Methanol concentration dynamics

In the simulated sample, methanol's behavior closely repeats the pattern described in the literature: a noticeable portion remained present until the end of the last distillation stages.

#### 4.2.4. Isoamyl alcohol

As previously discussed, the head fraction should be separated from the heart also for the intense pungent, solvent, fusel/solvent aromas in the head to due to higher concentrations of isoamyl alcohol as well, which is identified as a key odor active compound in many spirits. These higher alcohols, including isoamyl alcohol, tend to appear in greater concentrations in the head than in the heart or tail fractions.

Representing the Type 7 compound, isoamyl alcohol shows a sharp increase in concentration, including the characteristic "hook", at the very beginning of the distillation. This behavior can likely be explained by the fact that isoamyl alcohol has a relatively high boiling point compared to the temperature of the mixture, causing its evaporation to begin with a slight delay. This early peak is clearly reflected in the graph (Figure 22)

generated by the DT simulation. After this initial spike, its presence gradually decreases as the process continues.

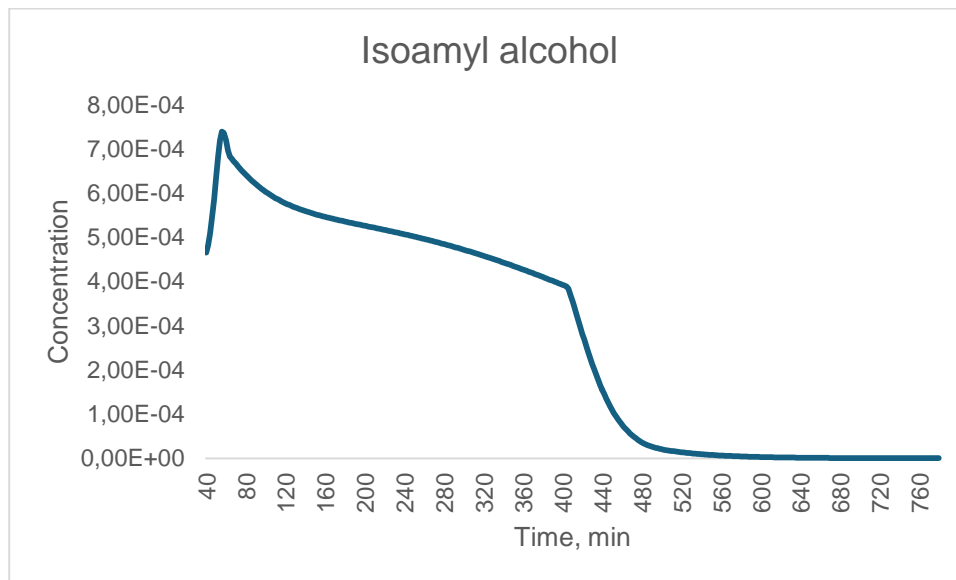


Figure 22 Isoamyl alcohol concentration dynamics

Isoamyl alcohol significantly affects the aroma and taste of the spirit. In small amounts, it contributes to the aromatic complexity, but at high concentrations (>3500 mg/L), it is characterized by penetrating odors that mask the aromatic finesse, consequently, it must be carefully controlled.

#### 4.2.5. Furfural

Furfural, being a heterocyclic aromatic aldehyde, demonstrates the behavior completely different from that of other aldehydes. The literature sources [41] illustrate a much lower concentration at the start, but it rises to the tail fraction as ethanol in the distillate begins to drop.

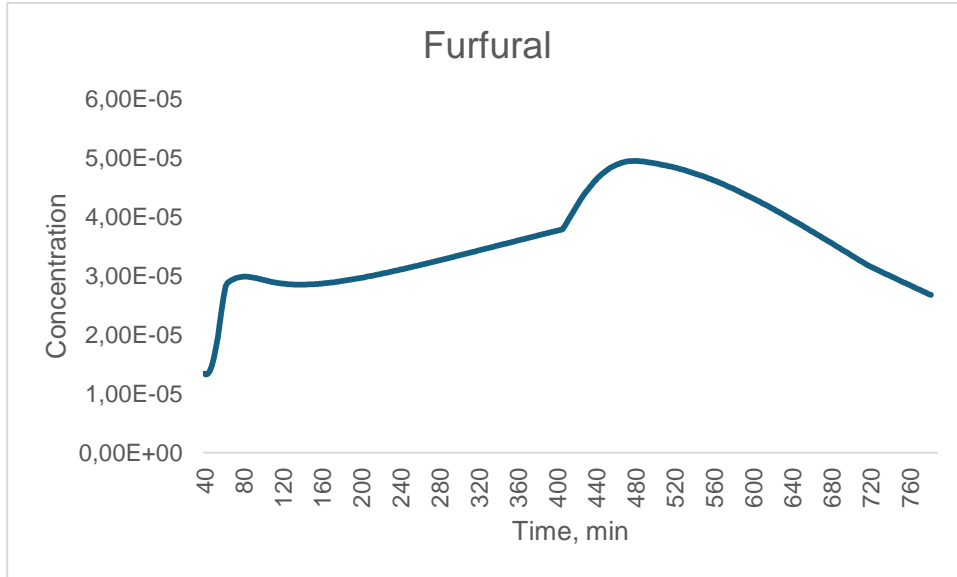


Figure 23 Furfural concentration dynamics

In other words, Furfural has a very low absolute volatility at high ethanol content, becoming more volatile when ethanol decreases and being extracted in the distillate only at the end of the heart extraction, and it matches the results from the DYNOSIM® simulation.

# 5 Conclusions and further steps

## 5.1. Conclusions

This work presents a simulation of the second distillation stage in Cognac production using a batch distillation model. The results show that the model can effectively describe the key stages of the process—from cold startup and reflux, to the separation of distillate fractions, and final shutdown.

Overall, the system performs well in reproducing the key steps of traditional Charentais alambic distillation, reaching target set points within a reasonable timeframe and maintaining process conditions close to those observed in real operations.

The results of the simulation show that the commercial software DYNOSIM® is capable of effectively reproducing process conditions, making it a useful tool both for analyzing distillation performance and predicting product outcomes.

The behavior of volatile compounds examined in this study is well reproduced by the DYNOSIM® simulation, closely reflecting their patterns in the real Charentais alambic distillation. This allows for accurate tracking of how these substances behave at different stages of the process.

Given that the distillation process is one of the most cost intensive steps in beverage production, and the composition of volatile compounds greatly affects the quality of the spirits, the ability to study and optimize this process through simulation instead of physical trials offers significant opportunities to reduce costs and improve quality in this very traditional, and to some extent elite field of food production.

## 5.2. Further Steps

This thesis examined the second distillation, a precise and narrowly defined stage in the Cognac production process.

To continue the current thesis work, several potential steps and improvements can be suggested.

The most logical next step would be to simulate the first stage of distillation — from raw wine to *brouillis*. Beyond Cognac, the same approach could be applied to other types of spirits such as whiskey, rum, all types of brandy, and others. Given the availability of experimental data for many of these beverages, which have been referenced in this thesis work, dynamic modeling tools offer a promising way to study and optimize these traditional processes.

Further work could include expanding the list of studied congeners to determine the sensitivity limits of the simulation software in terms of the minimum concentrations it can accurately model. This would allow for a more realistic formulation of the distillate mixture and contribute to a better understanding of the complexity and practical challenges involved in Cognac production. An interesting direction could be to enable chemical reactions in the Tower column to study how certain components, furfural, for example, transform during the distillation process.

Another potential improvement could be the implementation of a control system for more accurate separation of the fractions that could rely on real-time ethanol measurement to automatically operate the corresponding valves. This could make it possible to fully digitize the distillation process in Cognac production, opening up opportunities for better control and regulation without using expensive equipment and raw materials. It would also help identify key optimization steps and contribute to improving the quality of this “noble” drink.

Implementing heat-saving techniques, such as wine preheating, as used in the real Charentais alembic, could be another valuable direction for future development. This approach has the potential to improve overall process

efficiency by integrating energy systems that aim to minimize energy use and associated costs [19].

Furthermore, the heat and time regimes of the entire process and of each step could be optimized, which would potentially lead to increased energy efficiency, cost savings and increased productivity.

With these future steps, research in the field of batch distillation for the production of alcoholic beverages can be significantly expanded, resulting in better performance, energy efficiency, and sustainability.

A deeper understanding of the complex interactions in the distillation processes may ultimately drive innovation in winemaking and distilled spirits production, industries that still remain highly traditional, customary and handed down from generation to generation.

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