

# Pilot Design of a Distillation Column to Obtain Isoamyl Alcohol from Fusel Oil

Douglas Ramos Alves<sup>a\*</sup>, Jose Roberto Delalibera Finzer<sup>b</sup>, Edilberto Pereira Teixeira<sup>c</sup>

<sup>a,b,c</sup>University of Uberaba, PPGEQ, Airport Campus, Av. Nenê Sabino, 1801, Bairro Universitário, Uberaba-MG:38.055-500, Brazil

<sup>a</sup>Email: [douglasramos.eng@outlook.com](mailto:douglasramos.eng@outlook.com), <sup>b</sup>Email: [jose.finzer@uniube.br](mailto:jose.finzer@uniube.br), <sup>c</sup>Email: [edilberto.teixeira@uniube.br](mailto:edilberto.teixeira@uniube.br)

## Abstract

Brazil stands out internationally in the production of biofuels, especially ethanol, and its importance is commonly the subject of several studies. In the sugar-alcohol sector, the distillation process is essential for the good quality of the final product. However, from this operation, a residue that does not have high economic value in its natural state, the fusel oil, is also produced. The present work aims to design a pilot distillation column to obtain the isoamyl alcohol present in this by-product to add economic value to it. The McCabe-Thiele method was selected for dimensioning the distillation column, and the main results consisted of a distillation efficiency of 36%, using 8 trays, and a purity of isoamyl alcohol analyzed by chromatography of 92,2 %. Therefore, the work presents an alternative for the use of this important by-product.

**Keywords:** Fusel oil; distillation; isoamyl alcohol.

## 1. Introduction

For over 40 years, Brazil, fostered by public programs to encourage ethanol production such as Proálcool launched in 1975, has been a forerunner in the production process of this fuel, which makes the sugar and ethanol sector one of the main pillars of national agribusiness. The frequent stimulus granted in this area of agroindustry is because Brazil is the world's largest producer of sugarcane due to its favorable soil and climate conditions, in addition to large areas for growing the crop. According to the Brazilian crop monitoring report issued in April 2018, around 633.3 million tons of sugarcane and 27.8 billion liters of ethanol were produced in the 2017/18 crop [1]. The sugar-alcohol field produces several by-products and, therefore, is an area in which projects for the use of a residue can be viable both from economic and socio-environmental point of view. One of these residues is fusel oil and, from its distillation, isoamyl alcohol is obtained, which can be used as an organic solvent, flavoring or in the cosmetic industry [2].

---

\* Corresponding author.

In view of the current destination of fusel oil in ethanol plants, which is sold in its raw form and, therefore, has a low market price or is simply discarded inappropriately, causing environmental problems, the distillation column design, presented in this work, seeks to add value to the residue, in addition to proposing a sustainable alternative to it.

The present work has a general objective of designing a pilot distillation column to process the fusel oil and the specific objectives of applying distillation concepts, verifying the column efficiency, and identifying the products through chromatography analysis.

### ***1.1. Fusel oil***

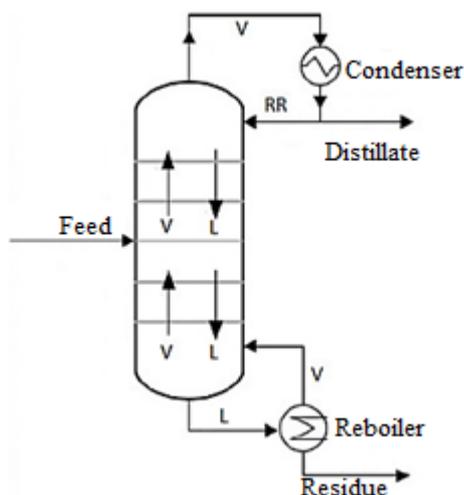
The term fusel oil (from the original Finkel) has German origin and means bad, weak, inferior. Currently, this term is widely used to indicate the mixture of alcohols, mainly higher, obtained in various stages of ethyl alcohol purification, representing the least volatile fraction of this process [3]. Its characteristics are a viscous liquid, with a slightly yellowish color and an unpleasant odor, and it has an average volume of production estimated in 2.5 liters for every 1000 liters of alcohol produced [4].

Higher alcohols are those with a molar mass greater than that of ethanol, that is, they have more than two carbon atoms in their molecule, such as the alcohols n-amyl, n-butyl, isopropyl, isobutyl, and isoamyl. The last is the compound present in greater quantity in the fusel oil [5].

Both the quality and the quantity of fusel oil generated during the distillation process depend on the reaction conditions in which the fermentation took place, the preparation of the broth to be fermented, and the method of removing the fusel oil [3].

### ***1.2. Distillation***

A distillation column, as shown in Figure 1, is formed by a cylindrical tank in a vertical position, whose material used in its construction depends on some parameters, such as the substances to be separated, pressure, and process temperature [6]. The column is designed to allow greater contact between the phases so that there is a greater transfer of heat, and mass between them. There is also a reboiler at the bottom of the column responsible for causing the vaporization of the mixture and a condenser at the top that performs the condensation of the vapor. A vessel can also be connected to the condenser to store the condensed vapor from which some of it is pumped back to the top of the column as reflux.



**Figure 1:** Scheme of a distillation column.

The interior of the distillation column can contain trays with different configurations, or the column can be filled with some material. The filling material has the same purpose as the trays, which is to increase the contact between the liquid and vapor phases [6]. There is also a classification called empty column, in which the Vigreux column can be cited as an example.

A common configuration of a tray distillation column is composed of the active and the areas of spillways, in addition to the dead and calm zones. The active part is the one where there is the maximum interphase contact of the mixture. It is the area where most of the heat and mass transfer occurs. Devices such as valves, bubblers or orifices are placed in this location and corresponds to the cross-sectional area of the column, except for the area of the spillways [7]. The calm regions are the areas between the liquid inlet spillway, and the first row of orifices, and between the outlet spillway and the last row of orifices. The dead areas are the parts of the tray that are close to the inner wall of the column and that do not participate in the mass exchange system [8].

### **1.3. McCabe-Thiele distillation method**

The “McCabe-Thiele” method is used for dimensioning distillation columns of binary mixtures of substances. It uses heat as a means for phase separation and is developed using an equilibrium diagram between liquid and vapor, called equilibrium curve [9].

In addition to the equilibrium curve, the McCabe-Thiele distillation method comprises the following items: a reference line with a 45° angle, the operating lines of the upper section of the feed (rectification), the operating line of the lower section of the feed (depletion), and the “q” line, or power line, that corresponds to the conditions of the power stage [7].

There are several methods for separating mixtures, but distillation is the most used procedure for separating liquids. The distillation method requires a study of the vapor-liquid phases in equilibrium. For the distillation step to occur with greater efficiency, it is important that all thermodynamic analyzes be based on data of the

greatest possible reliability, the main one being the Liquid-Vapor Equilibrium (ELV) data [10].

### 1.4. Equilibrium Curve

Some equilibrium data are needed to initiate the dimensioning process of a distillation column using the McCabe-Thiele method. This is illustrated in figure 2, where  $x$  and  $y$  are the molar fractions of the most volatile component in the liquid and vapor phases, respectively.

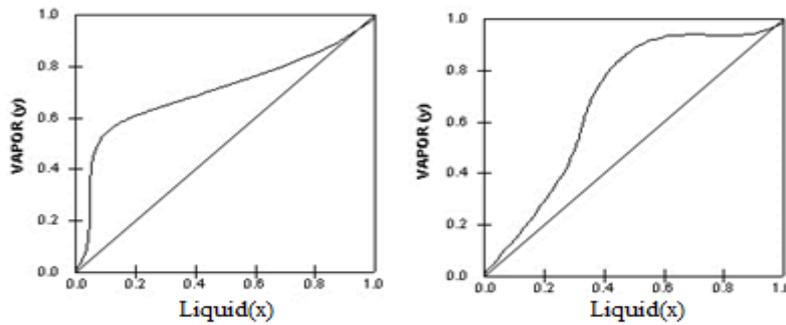


Figure 2: Systematic models of equilibrium curves.

## 2. Methodology

Some fusel oil of the 2017/2018 harvest supplied by CMAA - Companhia Mineira de Açúcar e Alcool, installed in the city of Uberaba, MG, Brazil, was used in this work. It was processed in the Laboratory of Unit Operations of the Graduate Program in Chemical Engineering at the University of Uberaba, Brazil.

### 2.1. Distillation of fusel oil in a pilot column

The designed equipment has a supply tank with a capacity of 10 liters of fusel oil. The pilot distillation column consists of a series of peripherals required for batch, or continuous operation, as shown in Figure 3. Table 1 identifies the components of the distillation column.

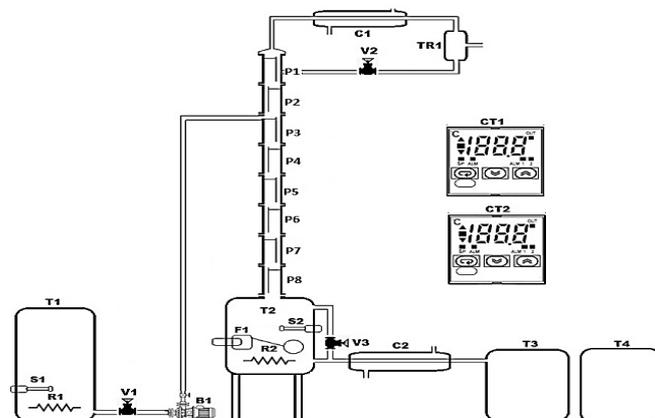


Figure 3: Pilot distillation column of fusel oil.

**Table 1:** Distillation column components.

<b>SIMBOLS</b>	<b>DESCRIPTION</b>
P1, P2, P3, P4, P5, P6, P7 e P8	Trays of the distillation column
T1, T2, T3 e T4	Storage tanks
V1, V2 e V3	Valves
C1 e C2	Condenser and evaporators
R1 e R2	Resistors
S1 e S2	Temperature sensors
CT1 e CT2	Temperature controllers
TR1	Recycle chamber
F1	Icos floater
B1	Arduino centrifuge pump

## 2.2. Experimental procedure

The feed tank T1 was filled with 10 liters of fusel oil and the controller CT1 was turned on. It was responsible for maintaining the temperature of the fusel oil in tank T1 at 80°C using sensor S1. After reaching this temperature, the centrifugal pump B1 was activated, and pumped the heated fuse oil to the feed tray P3.

Subsequently, the liquid flowed to the base of the heated column in the space over resistor R2. Immediately, by the action of the float F1, the resistor R2 was activated and heated the oil up to 125°C. This operation was performed by the controller CC2, and by the sensor S2. The column feed was kept constant, and all heat exchangers were fed with water approximately at 30°C, during the entire distillation step. The 2:1 recycle ratio was controlled by the valve V2 of the recycle drum TR1.

After the total feed of the fusel oil, the distillation including the recycling operation took place for 20 minutes. After the condensation of the vapors, the valve V3 was opened and all the isoamyl alcohol from the base of the column was sent to the tank T3, and a sample was taken from it for characterization. In the T4 tank, all the compound or by-product of the top product of the column with a boiling point lower than isoamyl alcohol was sent.

## 3. Result

The fusel oil was qualitatively characterized, for the design of a binary distillation column of only ethanol and isoamyl alcohol. This is an approximation since there are some other substances in the fusel oil.

It was necessary to obtain the vapor-liquid equilibrium curve of the ethanol-isoamyl alcohol system for designing the column using the McCabe-Thiele method. The curve was proposed by Silva [11], using EMSO software.

To test the thermodynamic models (UNIFAC-Dortmund/SRK), some experimental ELV data from the binary mixture of the components present in the column load, ethanol-isoamyl alcohol, were used. They have been compared with the results of the ELV calculation obtained with EMSO. The experimental data were obtained from the Dechema collection [12].

After obtaining the equilibrium curve (Figure 4), the necessary calculations to determine the feed line could begin. To prevent heat losses, the column feed stream was previously heated at 80°C, and with a composition of 30% steam and 70% liquid. That corresponds to a value of 0.7 for “q”, that stands for the moles of liquid flowing to the depletion section as a result of each mole of feed. This value agrees with that proposed by the author [13], which shows that the physical state of the feed is a mixture, in equilibrium, of the liquid and vapor phases, with the value of “q” between 0 and 1.

After selecting the value of “q”, some relevant calculations have been performed to plot the column operation lines. The ethyl alcohol composition in the fusel oil returned the value of  $x_F$  as 0,30.

For the construction of the supply line, it was necessary to find the value of its linear coefficient, which is expressed by the second part of Equation (1), obtaining the value of 2.33 for the angular coefficient.

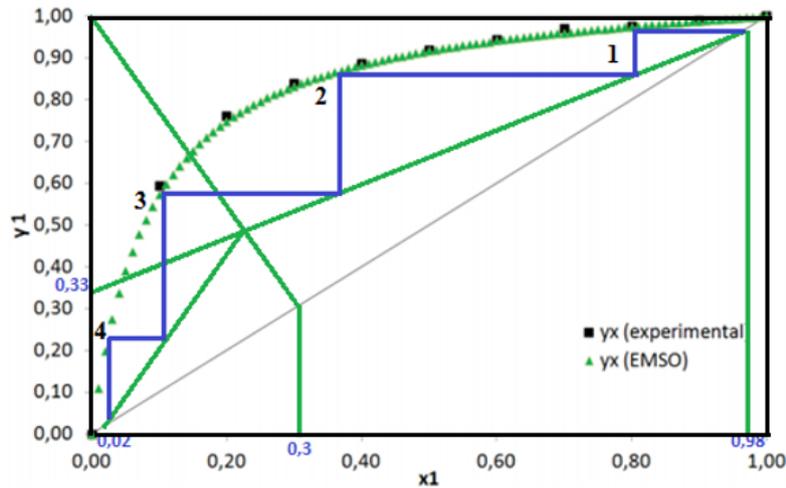
$$y = -\frac{q}{1-q}x + \frac{x_F}{1-q} \quad (1)$$

The result obtained allows locating the feeding line of the column in the McCabe-Thiele graph and, consequently, obtaining in which of the trays the feed would be performed, in the case of the ideal system.

The recycle ratio  $R_D = 2$  was selected by tentative in a non-optimized procedure, although it could increase the distillation costs. An optimization procedure would be more appropriate for a larger unit. Consider that the distillate composition of 98% ethanol is selected ( $X_D = 0.98$ , and  $X_B = 0.02$ ). With these parameters the purity of the isoamyl alcohol should present a commercially adequate value and it would not be necessary to design a column with many trays if the background product concentration were lower than the selected value. Thus, the linear coefficient representing the upper operating line is given by Equation (2) [9]:

$$\text{Linear Coefficient} = \frac{x_D}{R_D + 1} = \frac{0,98}{2 + 1} = 0,33 \quad (2)$$

With this coefficient, the upper operating line was located. The composition of the bottom product is 2% ethanol. With the values of the feed and the upper operating lines, together with the molar fractions of ethanol in the feed, top, and bottom streams, the McCabe-Thiele graph was prepared for the specification of the distillation column [9], represented by Figure 4:



**Figure 4:** McCabe-Thiele development of the distillation column design in the extraction of isoamyl alcohol from fusel oil.

### 3.1. Mass balance

The column was designed to produce 5 liters per hour of ethyl alcohol, as discussed earlier. The temperature at the top of the column is 112°C, but ethanol condenses at a temperature of 78°C, whose density is 0.789 g/cm<sup>3</sup>. Then, the amount of ethanol produced (D stream) is given by Equation (3).

$$D = \frac{Q \times \rho}{M} = \frac{5000 \frac{\text{cm}^3}{\text{h}} \times 0,789 \frac{\text{g}}{\text{cm}^3}}{46 \frac{\text{g}}{\text{mole}}} = 85,76 \frac{\text{mole ethanol}}{\text{h}} \quad (3)$$

At the top of the column, the amount recycled is given by the recycle ratio,  $R_D$ , established as 2, then the flow that returns to the column for enrichment was quantified, according to Equation (4):  $L = 171,52 \frac{\text{ethanol mole}}{\text{h}}$

$$R_D = \frac{L}{D} = 2 = \frac{L}{85,76} \quad (4)$$

The flow V, which is the vapor stream at the top of the column, is obtained as shown in Equation (5):

$$V = D + L = 85,76 + 171,52 = 257,28 \frac{\text{mole}}{\text{h}} \quad (5)$$

The feed flow F, is obtained from the global mass balance, given by Equation (6):

$$F = D + B \text{ then: } B = F - 85,76 \quad (6)$$

To go on with the calculation, a partial mass balance was carried out, as in equation (7).

$$x_F \cdot F = x_D \cdot D + x_B \cdot B \quad (7)$$

$$0,3F = (0,98 \times 85,76) + 0,02(F - 85,76)$$

$$F = 294,04 \frac{\text{mole}}{h}$$

The value of B represents the flow of the bottom product, which is mostly made up of isoamyl alcohol, and is given by equation (8):

$$\bar{B} = F - 85,76 \quad (8)$$

$$\bar{B} = 294,04 - 85,76$$

$$\bar{B} = 208,3 \frac{\text{isoamyl alcohol mole}}{h}$$

### 3.2. Number of column stages

According to Figure 5, the theoretical number of trays would be equal to four, three in the rectification section, and one in the depletion section. Feeding in the ideal system would occur on the third tray.

To determine the number of actual trays in the column, the empirical correlation described by the author [14] was used, where the hydraulic parameters of the trays are disregarded, considering only the viscosity ( $\mu$ ) of the most volatile component, the relative volatility ( $\alpha$ ) of the mixture, and the average temperature of the entire column.

The average temperature is given by the square root of the product of the maximum, and the minimum column temperature [15], as in equation (9).

$$\bar{T}_{column} = \sqrt{T_{max} \cdot T_{min}} \quad (9)$$

$$\bar{T}_{column} = \sqrt{T_{isoamyl\ ethanol} \cdot T_{ethanol}}$$

$$\bar{T}_{column} = \sqrt{130 \cdot 78}$$

$$\bar{T}_{column} = 100,7^{\circ}C$$

The viscosity of ethyl alcohol at the average column temperature was 0.32 cP. On the other hand, the relative volatility ( $\alpha$ ) is the relationship between the vapor pressure of the components of the mixture that vaporize. The higher the value of  $\alpha$ , the easier it is to separate the components. In calculating volatility, the vapor pressure of the most volatile is entered in the numerator and vapor pressure of the least volatile in the denominator [16]. In the case of the mixture under study, the relationship is according to Equation (10):

$$\alpha = \frac{P_{v\ ethanol}}{P_{v\ isoamylico}} \quad (10)$$

The vapor pressure of ethanol at the average column temperature is 225.979 kPa [17], and the pressure of isoamyl alcohol is 18.665 kPa [18]. Therefore, the relative volatility is:

$$\alpha = \frac{225,979 \text{ kPa}}{18,665 \text{ kPa}} = 12,11$$

As the number of actual trays depends on the column efficiency ( $\eta$ ), O'Connell [14] establishes that it can be calculated by Equation (11): then  $\eta=0.36$ .

$$100 \times \eta = \frac{50}{\sqrt[4]{(\alpha \times \mu)}} = \frac{50}{\sqrt[4]{(12,11 \times 0,317375)}} \quad (11)$$

According to the empirical method of O'Connell [14], the column has an overall efficiency of 36%, which corresponds to a lower value than most columns, which generally operate from 65 to 80% efficiency [16].

The calculation of the number of actual trays is closely related to the column efficiency and to the theoretical number established in the McCabe-Thiele method. However, initially, a unit was subtracted from the theoretical number since this represents the evaporator (reboiler) whose efficiency approaches the ideal condition, given by Equation (12) [19]:

$$NPT = \text{Number of trays} - 1, \quad \text{then } (4 - 1) = 3 \quad (12)$$

With this result, the actual number of trays could be calculated by Equation (13)

$$NPR = \frac{NPT}{\eta} = \frac{3}{0,36} = 8,33 \text{ trays} \quad (13)$$

That corresponds to the integer number of eight trays for the column. The diameter, and the height of the column could be calculated as 5, and 112 cm, respectively. The number of 82 holes in each tray was also calculated [19]

### 3.3 Distillation in tray column

For the assembly of the distillation column, the perforated trays were connected to flanges. The containers for feeding, and for residue collection were mounted separately to give the column more mobility, leaving it coupled only with the container for obtaining isoamyl alcohol, with the condenser, located on top, and the heat exchanger to cool the bottom product. In the electrical part, the required resistors were installed at the bottom of the supply container and at the base of the column where a temperature sensor was also installed. The temperature controller setpoint was also configured, obtaining an actuation range of 125°C, with a variation of  $\pm 5^\circ\text{C}$ . A contactor was used to operate the resistor by the controller. A view of the distillation column is available in Figure 5.



**Figure 5:** The distillation column already assembled.

Then, the distillation of the fusel oil was carried out. Ten liters of fusel oil, with an initial temperature of 24°C, were added to the feed container, where it remained for 10 minutes to reach a temperature of 80°C. Then, a pump with a flow capacity of 1.3 l/min was activated, transferring the fusel oil from the container into the column through the third tray, defined by design as ideal for feeding. A display attached to the base of the column allowed us to visualize the required amount of fusel oil and, as soon as it was supplied, the pump stopped working. The column was designed to be operated continuously, but it was not possible because of the small amount of available oil. This topic can be carried out in a future work.

Distillation was carried out and, after about 10 minutes, the temperature was in the range of 85°C and ethyl alcohol began to rise through the trays, starting the separation operation. The temperature rise ceased for a period when the largest volume of ethanol was collected. Then, with the eventual increase in temperature, compounds present in lower concentrations were also evaporated and purified the product of interest.

When the temperature reached 125°C, the valve of the 2:1 recycle drum was opened and the controller enabled operation between 120 and 130°C for 20 minutes. After this time, the distillation was stopped for 10 more minutes waiting for the vapors present inside the column to be completely condensed. After 50 minutes of operation, the products finally obtained could be quantified. All these results could be obtained by applying the authors experience in the operation of industrial distillation columns.

The amount of 5 liters established in the column design could be extracted from the top of the column in about 35 minutes of operation. It can be a result of the use of a high-power resistor. According to the energy balance, a resistor of 1.76 kW would be necessary to supply the thermal demand. Besides that, we decided empirically to install a resistor of 4.3 kW, allowing to obtain the designed top volume in a shorter time.

Considering the entire operating time, 5.83 liters of the upper product could be obtained, which showed a good

performance of the column since this volume represents about 60% of the residue present in the fusel oil. As ethanol concentrations in industrial fusel oil vary, it is possible that, from another sample, this value would change.

Regarding to the bottom product, which is isoamyl alcohol, 3.94 liters were obtained, which represents, in volume, about 40% of the fusel oil. This volume demonstrates an increase when compared to the amount obtained by distillation in a Vigreux column, which can be justified by the more efficient temperature control and a greater interaction between the phases obtained in the tray column. As it contains a recycling system, the increase, or decrease of the recycling ratio could lead to obtaining an alcohol with different levels of purity. This is important for diverse industrial uses [5].

During the operation, two main difficulties were found, the first of which was the fact that the condenser was inefficient in cooling the top product to the temperature required in the project. It was possible to condense the steam, but it was at a temperature of 46°C and not at 25°C as had been specified, since the flow of water used in the condenser was about twice lower than that obtained in the energy balance, which would be 4 l/min. It was not possible to adjust the flow because the water source (lab faucet) was already turned on at maximum.

The second and main difficulty was related to the color of isoamyl alcohol, which was darker than the standard. The fusel oil sample also had a darker color than normal. As the product of interest, in this case, is the bottom product of the distillation, it was not possible to change the characteristic color of isoamyl alcohol, a fact demonstrated by Figure 6.



**Figure 6:** Samples of fusel oil and isoamyl alcohol.

After the distillation step, some physical chemical analyzes of the isoamyl alcohol were carried out. The pH was 5.27, a value below the standard that establishes the pH of the compound as neutral (value equal to 7). This more acidic pH can be explained by the pH of the fusel oil sample used for its extraction, which showed a value of 4.98. According to [20], this result can be justified because more acidic pH let the formation of higher alcohols, which are the substances that make up the fusel oil.

Density analysis was performed using a digital densimeter and returned the value of 0.817 g/cm<sup>3</sup>, a result that is very close to 0.810 g/cm<sup>3</sup> which the density obtained by Merck [21]. This value can be used as one of the

indicators that the product obtained is really the one of interest, thus evidencing a good distillation process.

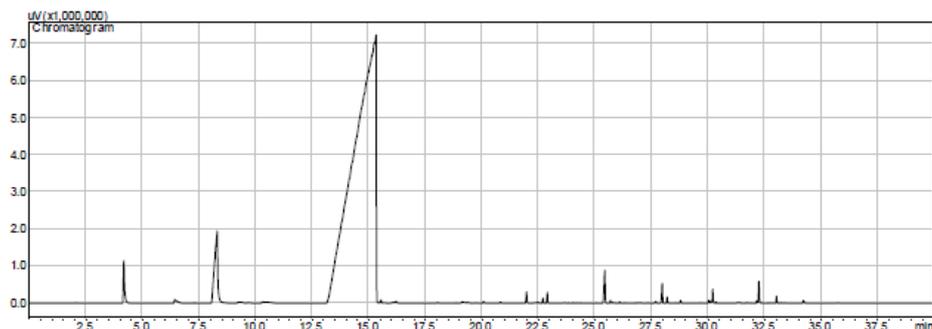
The product of this distillation was also subjected to a gas chromatographic analysis coupled to a mass spectrometer (GCMS-QP2010Ultra) brand Shimadzu, model GCMS-QP2010Ultra. The Gas chromatograph GC 2010-01 of the brand Shimadzu (model GC – 2010) where used to identify compounds carried on by the IPT laboratory as shown in Table 2:

**Table 2:** Analytic Chromatographic Results.

Item	Compound	Molecular formula	Content (%)
1	Ethanol	$C_2H_6O$	$1,3 \pm 0,1$
2	1-Propanol	$C_3H_8O$	$0,20 \pm 0,01$
3	Isobutanol	$C_4H_{10}O$	$3,8 \pm 0,1$
4	Isopentyl acetate	$C_7H_{14}O_2$	$0,09 \pm 0,01$
5	1- Butanol	$C_4H_{10}O$	$0,15 \pm 0,01$
6	2 Methyl-1-butanol (little) + Isopentanol	$C_5H_{12}O$	$92,2 \pm 0,4$
7	Ethyl hexanoate	$C_8H_{16}O_2$	$0,07 \pm 0,01$
8	1-Hexanol	$C_6H_{14}O$	$0,03 \pm 0,01$
9	Maybe 3-Hexen-1-oil	$C_6H_{12}O$	$0,02 \pm 0,01$
10	Ethyl octanoate	$C_{10}H_{20}O_2$	$0,15 \pm 0,01$
11	Ethyl decanoate	$C_{12}H_{24}O_2$	$0,48 \pm 0,03$
12	Isopentyl octanoate	$C_{13}H_{26}O_2$	$0,03 \pm 0,01$
13	2-Phenylethyl acetate	$C_{10}H_{12}O_2$	$0,02 \pm 0,01$
14	Ethyl dodecanoate	$C_{12}H_{24}O_2$	$0,18 \pm 0,01$
15	Maybe isopentyl decanoate	$C_{15}H_{30}O_2$	$0,05 \pm 0,01$
16	2-Phenylethanol	$C_8H_{10}O$	$0,03 \pm 0,01$
17	Probably nerolidol	$C_{15}H_{26}O$	$0,02 \pm 0,01$
18	Maybe dimethoxystyrene	$C_{10}H_{12}O_2$	$0,02 \pm 0,01$
19	Ethyl tetradecanoate (little)+octanoic acid	$C_{16}H_{32}O_2 + C_8H_{16}O_2$	$0,20 \pm 0,01$
20	Ethyl hexadecanoate	$C_{18}H_{36}O_2$	$0,02 \pm 0,01$
21	Decanoic acid	$C_{10}H_{20}O_2$	$0,31 \pm 0,01$
22	Farnesol	$C_{15}H_{26}O$	$0,06 \pm 0,01$
23	Dodecanoic acid	$C_{12}H_{24}O_2$	$0,05 \pm 0,01$
24	Unidentified compounds	-	$0,50 \pm 0,07$

As shown in Table 2, isoamyl alcohol appeared in major amount, with 92.2% of purity and these results are 95% reliable in average of 3 samples, and the limit of the quantification method is 0.01 %.

With the result of 92% obtained from the chromatograph, there is confirmation that the desired product was obtained and its purity is one of the highest. The rigid control of process variables was very important for this result to be obtained. With high purity, isoamyl alcohol can be used in the food and cosmetics sectors, in addition, there is an increase in market value, making it a more profitable product. The analytical peak of isoamyl alcohol can be seen in Figure 7:



**Figure 7:** Chromatogram of isoamyl alcohol.

Figure 7 shows the chromatogram of the LAQ 1101-20 material from the IPT laboratory. The chemical compound corresponding to the highest intensity peak of the retention time (is the time required after injection of the mixture into the column until that component reaches the detector)  $\approx 15$  minute, so it is isoamyl alcohol. In the organoleptic analyses, the only factor that disagreed with the forecast was the commercial color of the product, explained above, since the odor and density were adequate when compared to a standard. A variable that certainly has to be taken into account when proceeding with the method is the choice of raw material, that is, the fusel oil. None of the previous studies that address this issue had to deal with this darker coloration that existed in the sample used. As much as the other parameters were met, a deeper study is needed in relation to the color of both the sample and the final product in order to establish whether in the long term this aspect becomes a problem. The quality of the sugarcane or the distillation of ethanol may be some of the contributing factors for the browning of fusel oil.

#### 4. Conclusion

The development of the present work allowed an analysis of the fusel oil, and the aggregation of value to the by-product in the separation, and use of its major compound, isoamyl alcohol. Its use as an alternative source of income for the sugar and ethanol industry is promising. As one of the pillars of sustainable development, ethanol stands out in the field of biofuels, and now it can also be recognized by the applicability of fusel oil, since the separation of its constituents brings social, environmental, and economic benefits. The column design, using the McCabe-Thiele method, with a 5 cm diameter, 1.22 m height, and consisting of eight perforated trays proved to be effective in the distillation of fusel oil. The mass balance, carried out by relating the molar flows, was fundamental for the development of this project, since the amounts of intrinsic product, both from the top and from the bottom of the column, were quantified. Comparing the distillation of fusel oil in a Vigreux column of trays, a greater efficiency in the separation by the latter method could be verified. In the Vigreux column, 34.9 mL of isoamyl alcohol were obtained from 100 mL of fusel oil and in the tray column, 39.94 mL were obtained for the same amount of fusel. This can be explained by the greater interphase interaction provided by the perforated trays inside the column. Regarding the physicochemical parameters, most of them provided results like those found in the literature, differing only for the isoamyl alcohol pH. The analyzes were essential to characterize isoamyl alcohol. It is concluded that the main objective of the work, which is the design and construction of a pilot column for the distillation of fusel oil to obtain isoamyl alcohol, was achieved.

Furthermore, this by-product shows itself as a probable profitable alternative for the supply of an important raw material used in various industrial areas, adding value both to the fusel oil itself, and to isoamyl alcohol, thus contributing to the management of this waste.

## References

- [1] CONAB (National Supply Company) – Monitoring the Brazilian crop: sugarcane, v. 4 - 2017/18 Harvest, no. 4 - Fourth survey, Brasília, p. 1-73, April 2018. (In Portuguese language)
- [2] M.A.D. Oliveira, N.A. Barbosa and T.M. Giati. “Simulation of obtaining isoamyl alcohol from fusel oil via distillation” Monograph (Bachelor of Chemical Engineering), São Francisco University, Campinas, 2016. (In Portuguese language)
- [3] A.G. Patil, S.M. Koolwal and H.D. Butala. “Fusel oil: composition, removal and potential utilization” International Sugar Journal, v.104, n.1238, p.51-63, 2002.
- [4] E.R. Perez, D.R. Cardoso and D.W.Franco. “Analysis of alcohols, esters and carbonyl compounds in fusel oil samples” Química Nova, vol.24, n.1, p.10-12, fev. 2001. (In Portuguese language)
- [5] M.C. Ferreira. “Study of the fusel oil distillation process” Dissertation (Master in Food Engineering), Faculty of Food Engineering, State University of Campinas, Campinas, 2012. (In Portuguese language)
- [6] M.G. Silva. “Modeling and simulation of a distillation column for separating the reaction components of biodiesel in MATLAB” Completion of course work (Bachelor of Chemical Engineering), Department of Chemical Engineering, Federal University of Rio Grande do Norte, Natal, 2015. (In Portuguese language)
- [7] J.G. Alvares and B.D. Souza. “Design of a Didactic Distillation Column” Course Completion Work (Graduate in Chemical Engineering), Federal University of Alfenas, Poços de Caldas, 2013. (In Portuguese language)
- [8] J.N. Caldas, A.I. Lacerda, E. Veloso and L.C.M. paschal. “Torres' Interns: Trays and Stuffing” 2nd ed. Rio de Janeiro: Petrobras, Interciencia, 2007. (In Portuguese language)
- [9] J.D. Seader, E.J. Henley and D.K. Roper. Separation Process Principles: Chemicals and Biochemicals Operations, 3rd ed. Danvers: John Wiley & Sons, 2010.
- [10] J.C.S.L. Maciel. “Study of the vapor-liquid balance of the water+ethanol+ionic liquid system aiming at the separation of anhydrous alcohol” Dissertation (Master in Chemical Engineering), Graduate Program in Chemical Engineering, Federal University of Rio Grande do Norte, Natal, 2012 . (In Portuguese language)
- [11] J.I.S. Silva. “Dynamic simulation of the bioethanol distillation process in an equation-based simulator

(EMSO)” Dissertation (Master’s in Chemical Engineering), Graduate Program in Chemical Engineering, Federal University of São Carlos, São Carlos, 2012. (In Portuguese language)

- [12] J. Gmehling, U. Onken; J.R. Rarey-Nies. Vapor-liquid equilibrium data collection – aqueous systems. Dechema Chemistry Data Series, Vol 1, Part 1b. 1988.
- [13] A. Moriizumi. “Application of the McCabe-Thiele methodology in the modeling of a distillation column using Excel software” Monograph (Graduate in Chemical Engineering), School of Engineering of Lorena, University of São Paulo, Lorena, 2012. (In Portuguese language)
- [14] H.E. O’Connell. “Tray efficiency of fractionating columns and absorbers. Transactions of the American Institute of Chemical Engineers”, 42, pp. 741–775, 1946.
- [15] C. Magagnin, D.B. Machado, K. Campos, L.M. Sandrini and T. Oliveira. “Design of a Distillation Column” Report. Academic Unit of Technological Sciences, University of the South of Santa Catarina, Florianópolis, 2017. (In Portuguese language)
- [16] F.D. Mayer. “Development of distillation technology appropriate for the production of alcohol fuel on a small scale” Dissertation (Master in Process Engineering), Graduate Program in Process Engineering, Federal University of Santa Maria, Santa Maria, 2010. (In Portuguese language)
- [17] DB Dortmund. “Thermophysical Data for Process Design” Internet: <http://ddbonline.ddbst.com/AntoineCalculation/AntoineCalculationCGI.exe> [Mar. 16, 2021].
- [18] R. Stenutz. “Tables for chemistry” Internet: <http://www.stenutz.eu/chem/solv6.php?name=isoamyl+alcohol> [Mar. 16, 2021].
- [19] W.L. McCabe, J.C. Smith and P. Harriot. Unit operations of chemical engineering. 5th edition New York: McGraw Hill College, 1993.
- [20] L.E. Gutierrez. Production of higher alcohols by *Saccharomyces* strains during alcoholic fermentation. *Scientia Agricola*, v.50, n.3, p.464-472, Oct/Dec 1993. (In Portuguese language)
- [21] M. Merck. Chemicals for Industries. Chemical Information Sheet: Isoamyl alcohol. 2014.

### **Acknowledgement**

The authors thank the Fundação de Amparo à Pesquisa de Minas Gerais – FAPEMIG and UNIUBE for the support given to the development of this research.