

FUSEL OIL INFLUENCE IN ETHANOL DISTILLATION

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Abstract – The components of fusel oil form azeotropes with water, resulting in challenges in obtaining hydrous ethanol fuel (HEF) as well as higher investment and operating costs. The aim of this study was to evaluate the influence of fusel oil during HEF distillation. Mixtures of ethanol (6.0 to 6.5 % m/m) and water (93.0 to 94 % m/m) containing different concentrations (zero to 0.5 % m/m) of 3-methyl-1-butanol, 2-methyl-1-propanol, propan-1-ol and propan-2-ol were distilled in a laboratory apparatus. The results obtained in total reflux demonstrated the possibility of obtaining HEF according to Brazilian legislation specifications, although the desired concentration was not reached during continuous operation. All experiments resulted in a high loss of ethanol in the bottom product across multiple operating conditions. The results demonstrated that while fusel oil does affect HEF distillation, it does not prevent the attainment of HEF that is in conformity with Brazilian legislation.

Keywords: fuel ethanol, distillation; fusel oil;

1. INTRODUCTION

Fusel oil refers to high-molecular-weight compounds, particularly higher chain alcohols, obtained through the distillation of fermented carbohydrates. Fusel oil possesses a dark green color and an acrid smell (Rasovsky, 1979). Its components originate from yeast metabolic processes during fermentation. The composition of fusel oil is influenced by fermentation medium conditions (presence of amino acids, assimilable nitrogen compounds, temperature, aeration and inoculum concentration) and type of inoculums (Webb and Ingraham, 1963; Borzani et al., 1981; Gutierrez, 1993; Walker, 1998; Patil et al., 2002). Additionally, the composition of fusel oil is influenced by the feedstock used to obtain the fermentation substrate (Brau, 1957; Webb and Ingraham, 1963). Webb and Ingraham (1963) demonstrated the presence of up to 97 different substances in fusel oil. The proportion of fusel oil in the fermented mash ranges between 0.1 and 0.6% (Küçük and Ceylan, 1998; Patil et al., 2001; Pérez et al., 2001; Lima et al., 2001; Amorim et al., 2005). Brau (1957) reported values between 0.1 and 1.1%. Fusel oil is removed during the distillation of fermented mash (Garcia, 2008). Its high boiling point, in the range of 130-132 °C (Webb and Ingraham, 1963), requires removal in less volatile fractions during distillation. In batch distillation (for the production of distilled spirits), the obtained fusel oil is diluted in the "tail" stream, with a high concentration of water and ethanol. In continuous distillation, withdrawal occurs in the lower portion of the rectification column. In addition to the aforementioned factors, the concentration of each substance in fusel oil depends on the way in which it is removed from the distillation column.

After being removed in the rectifying tower, fusel oil is cooled and washed with water to recover the residual ethanol. The fusel oil is then distilled to recover the products, mainly amyl



alcohol (3-methyl-1-butanol). Amyl alcohol is the primary component (approximately 60%) of fusel oil. The term amyl alcohol can be traced back to the first encountered fusel oil identified in the distillates obtained from starch (amylum) fermentation (Simmonds, 1919).

Regarding distilled spirits, the removal of fusel oil has served as an incentive to technological changes in various continuous distillers following the development of the Coffey still (or Patent still) in 1830 (Gaiser *et al.*, 2002; Russel, 2003). The Coffey still has a configuration that is very similar to current ethanol distillation towers to produce high grade ethanol. This still is divided in two sections: stripping and rectification. A modification of this type of still allowed for the removal of a side stream from the plates with the highest concentration of fusel oil. According to Patil *et al.* (2001), this removal is complex due to the volatility of fusel oil. When the ethanol concentration is low, the volatility of fusel oil is higher than that of ethanol and the fusel oil is trapped in the region of the column corresponding to the ethanol concentration range between 40 and 47 %m/m. The removed oil stream may or may not split into two liquid layers depending on the prevailing conditions at the point of fusel oil removal (mass and energy balance). If separation occurs within the column and below the point of removal, separation may become impossible. Unfortunately, it is not known when this phenomenon will occur during operating conditions.

From 2.0 to 6.5% of total feed ethanol should be purged to remove the fusel oil from the distillate (neutral distillate) (Valderrama *et al.* 2012). According to Batista and Meirelles (2011), in the distillation of ethanol for drinking, the concentration of higher alcohols in the distillate tends to increase at low flow rates of distillate and low reflux ratios. An analysis of the equilibrium curve of the ethanol/water/3-methyl-1-butanol system to a temperature of 25 °C indicates that there is a wide concentration range for which there are two phases, allowing the separation and removal of 3-methyl-1-butanol (Kadir *et al.* 2008). However, towards the minimum temperature in the tower (close to 78 °C, at the top of the rectifying section), the biphasic region becomes smaller, indicating a limitation in fusel oil separation.

The presence of fusel oil causes perturbation in the operation of ethanol distillation because the occurrence of "cyclic flooding" due to the formation of a second liquid phase (Kister, 1990). According to Shinskey (1984), the higher alcohols form azeotropes with water during the distillation, and the accumulation of these alcohols occurs until the achievement of the limit of solubility, forming a second liquid phase. This behavior generates instability in the distillation operation because these two liquid phases exercise their total vapor pressure. Therefore, the upward vapor flow doubles instantaneously, lowering the temperature in the column. This flooding usually carries the fusel oil to the top of the column. The column heating inlet may be raised if a temperature control is manipulating the boilup. In both cases the fusel oil tends to be purged at the top of the column, after that the distillation returns to normal operation in few hours or few days (Shinskey, 1984).

Russel (2003) reported that the presence of 3-methyl-1-butanol in the lower portion of the rectification column prevents ethanol condensation. This condensation reduces the tray efficiency and increases the ethanol concentration in the bottom product of that column. It has also been reported that rectification is unhindered above a certain stage. Another important point shown by Russel (2003) refers to the relative volatility of 3-methyl-1-butanol, which is higher than that of ethanol when the ethanol concentration is less than 40% v/v. In simulations developed by Gaiser *et al.* (2002), the peak concentration of 3-methyl-1-butanol coincides with an ethanol concentration of 20% m/m (approx. 25% v/v). The operation used in fusel oil removal during HEF distillation is the same as in beverages, although more rough. This removal usually involves side withdraws, that is cooled prior to phase separation in which the aqueous phase, containing ethanol and water, returns to the distillation column.

Therefore, is it really necessary to remove fusel oil from ethanol used for fuel? As previously mentioned, the removal of fusel oil entails multiple constructive modifications to the distiller (flow points, fusel oil removal column and ethanol recovery column). These additions lead to higher construction and operating costs. Currently, all major ethanol distilleries remove fusel oil during distillation. For ethanol production on a small scale, a simple, cost-effective distillation system is essential for economic feasibility. Mayer *et al.* (2013) presented a single column distillation system, performing tests with a hydroalcoholic solution (ethanol + water), to yield distillate according to the ethanol concentration in agreement with ANP. However, the influence of fusel oil was not evaluated in that study.

In addition, the Brazilian legislation that regulates the commercialization of fuel ethanol (ANP Resolution N° 07, dated February 9, 2011) makes no direct mention about the presence of fusel oil or its components in the HEF. This resolution states that the maximum permitted level for hydrocarbons is 3.0% v/v, analyzed according to the standard from ABNT NBR 13993. However, this standard only assesses the presence of gasoline in the sample, implying that the presence of any higher alcohols in HEF are tolerated. The Technical Regulation CNP-07/82 (IBP, 1987), from the former National Petroleum Council, defined that the maximum allowed concentration of higher alcohols would be 6.0% m/m; however, this regulation is no longer valid. The European Union allows the addition of up to 7% oxygenates in gasoline, including 3-methyl-1-butanol (Council Directive of the European Communities 85/536/CCE). Cataluña et al. (2008) presented comparative results of oxygenates as fuel in internal combustion engines, indicating that 3-methyl-1-butanol has a lower specific fuel consumption and realeses more power compared to pure ethanol. More recently, ANP Technical Notes 04- and 97-/2013/SBQ/CPT-101-DF states that the presence of higher alcohols in HEF do not affect engines performance. Furthermore, these Notes pointed out that fusel oil has a commercial price higher than ethanol. Thus, the removal of fusel oil in larger distilleries is an economical option, which is not true regarding small scale ethanol production.

The aim of this paper is to evaluate the influence of fusel oil in ethanol distillation using a column bench, as proposed by Mayer *et al.* (2013). These findings will provide operational information to support the development and improvement of a pilot still.

2. MATERIALS AND METHODS

2.1 Analytical procedures

<u>2.1.1 Apparatus</u>: To monitor distillation progress, the ethanol concentrations in the top and bottom products were analyzed using a digital density meter (Anton Paar DMA 4500 M) with temperature control and repeatability of 0.00001 g.cm⁻³. The digital density meter directly converts the density measurement to the concentration of ethanol with a typical accuracy of 0.025 %m/m. The method for analyzing the concentration of higher alcohols was gas chromatography, which was developed and applied using a Perkin Elmer Instruments model Auto System XL GC equipped with a flame ionization detector. The data acquisition was performed using the software Perkin Elmer Turbochrom 4.1. The GC operating conditions were as follows: injection temperature, 250°C; detector temperature, 170°C; Column: PE-1 Perkin Elmer, 30-m long, 0.53-mm i.d.; carrier gas flow, helium at a flow rate of 3 μ L.min⁻¹. The operating conditions were as follows: manual sample feed; split ratio, 1:1; attenuation, zero; oven temperature program: 70 to 150°C, with a heating rate of +5°C. min⁻¹. The flow rates of the flame gases for the FID were as follows: hydrogen, 45 μ L.min⁻¹; synthetic air, 450 μ L.min⁻¹.

<u>2.1.2 Reagents</u>: The feed in the distillation tests was prepared using 95% commercial-grade ethanol. The higher alcohol patterns were propan-1-ol 99.5% (Vetec), propan-2-ol 99% (Synth), 2-methylpropan-1-ol 99% (Vetec) and 3-methyl-1-butanol 98.5% (Vetec). The internal standard was



methanol 99% (Merck). Ethanol 99.5% (Neon) was used in the sample solutions for preparing calibration curves.

2.1.3 Procedures: The analytical curves were obtained via triplicate injections of higher alcohol solutions at concentrations of 0.5, 1.0, 2.0, 3.0, 4.0 and 5.0 % m/m with an internal standard of 5.0 % m/m methanol. All the samples were completed with ethanol. The distillation samples for the GC analyses were prepared by adding 95% distillation sample and 5% internal standard (methanol). For the GC analysis, the injected sample volume was 1.0 μ L. The concentration of higher alcohols was determined by measuring the area ratio of each analyte to methanol and comparing it to calibration curves (area ratio analyte/standard *versus* analyte concentration).

2.2 Distillation procedure

The experiments were conducted on distilling solutions of ethanol + water + higher alcohol, according to the data presented in Table 1. The concentration of higher alcohols was based on the data presented in Batista and Meirelles (2011). The concentration was overestimated to ensure that the experiments represent conditions with the maximum concentration of fusel oil compounds found in fermented mash.

	Water	Ethanol	Higher alcohol (%m/m)							
Solution			3-methyl-1-	Propage 1 ol	2-Methylpropan-1-ol	Dropon 2 ol				
			butanol	Flopali-1-01	2-wieuryipiopaii-1-or	F10pall-2-01				
1	93.5	6.0	0.5	-	-	-				
2	93.5	6.0	-	0.5	-	-				
3	93.5	6.0	-	-	0.5	-				
4	93.5	6.0	-	-	-	0.5				
5	93.5	6.0	0.3	0.1	0.05	0.05				
6	94.0	6.0	-	-	-	-				

Table 1 – Composition of the six solutions with and without higher alcohols (%m/m)

The experiments were performed in a continuous distillation bench column, as proposed by Mayer *et al.* (2013), located in the Laboratory of the Process Engineering Post-graduate Program at Federal University of Santa Maria. This bench column is characterized by the use of vigreux and packing in sections of stripping and rectification, respectively. Each experiment was divided into two distinct stages. In the first step, distillation was performed at total reflux to verify the maximum concentration obtained by the distiller. Five liters of feed, according to Table 1, were introduced into the reboiler of the distillation apparatus to start the operation, which lasted until the temperature of the top vapor stabilized. Samples were collected in 10 minute intervals.

The second step consisted of continuous solution feeding (as shown in Table 1) at a rate of 3.206 kg.h^{-1} . The flow rates of the distillate and bottom products were 0.209 kg.h^{-1} and 2.997 kg.h^{-1} , respectively, to maintain a distillation performance of approximately 92%. The tests required an average of three hours, with ninety minutes dedicated to equipment heating and stabilization. Samples were collected every 10 minutes after stabilization of the concentration of the top product. More information regarding the features and operation of the distiller can be found in Mayer *et al.* (2013).

3. RESULTS AND DISCUSSION

The analysis of fusel oil interference in the distillation of ethanol was divided into two steps. The first step was the assessment of the interference against the ideal operating conditions



represented by the total reflux. The accumulation of fusel oil was visually observed in the region of the column corresponding to 75% m/m of ethanol. The concentrations of the top products in the six experiments in total reflux are presented in Table 2. The concentration of higher alcohols in the top product has a direct relationship with its boiling point because the lower the boiling point of the higher alcohol the higher its concentration in the top product. In addition, the experiments that not achieved the minimum ethanol concentration for top product were more affected by the formation of azeotrope between water and higher alcohols.

Higher alcohol (% m/m)										
Solution	Ethanol (%m/m)	3-Methyl-1-	Propan-1-ol	2-Methylpropan-1-ol	Propan-2-ol					
		butanol	riopun r or	2 Monypropun 1 of	riopuli 2 or					
1	89,46	0.63	-	-	-					
2	91,40	-	1.39	-	-					
3	89,98	-	-	0.64	-					
4	92,98	-	-	-	5.00					
5	92,67	0.00	0.04	0.14	1.01					
6	92,10	-	-	-	-					

Table 2 - Composition of samples at total reflux operation

From the experimental data, it can be observed that solutions 4 and 5 had ethanol concentrations greater than the minimum (92.5% m/m) required by ANP Resolution No. 07/2011. The other experiments had ethanol concentrations below that limit, and the minimum concentration was obtained in the experiment conducted with solution number 1, indicating that the presence of 3-methyl-1-butanol in the feed at concentrations greater than 0.3% (concentration in solution number 5) may compromise the separation of the ethanol/water mixture. This statement is plausible because other compounds (propan-1-ol and 2-methylpropan-1-ol) were present in solution number 5, which may result in synergistic behavior among the higher alcohols.

Experiments 4, 5 and 6 also demonstrate that when the number of separation stages is adequate (in this case the number of separation stages was infinite), it is possible to attain the minimum allowable limit in the presence of propan-1-ol and propan-2-ol only. It is important to note that the concentrations of higher alcohols used in the experiments were overestimated in order to represent an extreme situation because fermented mash concentration can vary widely. The second set of experiments aimed to evaluate the behavior of the distillation during continuous operation. The data obtained for the average concentration of ethanol and the reflux ratio for each of the six experiments are shown in Table 3. None of the tests obtained an ethanol concentration in the top product of greater than 92.5 % m/m, indicating that the number of separation stages in the distillation column is insufficient. The reflux ratio measured during the experiments was situated close to 3.0, which is the highest value obtained in a pre-flooding condition (loading point). With respect to the total reflux operation, the concentration of higher alcohols in the top product followed the boiling point, particularly propan-2-ol (4.95 % m/m). The concentration of propan-2-ol increases the HEF enthalpy of combustion by approximately 6.0%, suggesting an advantage for the presence of higher alcohols.

The concentration of ethanol in the bottom product was higher than that recommended by Meirelles and Batista (2011), 0.02% m/m. Mayer et al. (2013) indicated that 0.5% m/m would be the maximum recommended concentration due to the excessive loss of ethanol in the bottom product. The high concentration of ethanol in the bottom product is possibly due to the influence of higher alcohols, as cited by Russel (2003). Note that solution number 6 (ethanol + water) also showed a high concentration of ethanol in the bottom product. This finding can be explained by the fact that the tests were initially fed through the reboiler without sufficient time for stripping. This effect is shown in Fig. 1.b. A gradual decrease in the concentration of ethanol in the bottom



product was observed during distillation. This behavior indicates that a longer distillation processing and / or a greater number of stripping stages could result in an ethanol concentration that is in compliance with the maximum allowable limit. According to Table 3, the concentration of higher alcohols in the top product was greater than in the bottom product, indicating that these alcohols tend to concentrate in the rectification section. An analysis based on mass balance suggests that there may be an accumulation of higher alcohols in the column, influencing the separation of ethanol/water. This may require a longer duration experiments.

Table 3 – Mean concentration (%m/m) for the six tests in continuous operation (top and bottom products)

Solution .	Reflux ratio		Ethanol			3-Methyl-1- butanol Prop		Propa	Propan-1-ol		2-Methylpropan- 1-ol		Propan-2-ol	
	Average	σ^{a}	Тор	σ	Bottom	σ	Тор	Bot. ^b	Тор	Bot.	Тор	Bot.	Тор	Bot.
1	3.3	0.32	89.26	1.57	1.99	0.90	0.14	0.11	-	-	-	-	-	-
2	2.7	0.41	90.50	0.55	2.84	1.15	-	-	0.74	0.13	-	-	-	-
3	2.9	0.30	88.47	1.18	2.83	0.72	-	-	-	-	0.19	0.06	-	-
4	3.6	0.21	90.50	0.58	2.28	0.96	-	-	-	-	-	-	4.95	0.00
5	3.0	0.30	90.76	1.35	2.79	1.02	0.00	0.00	0.11	0.00	0.04	0.00	1.79	0.00
6	2.9	0.21	92.44	0.17	1.33	0.65	-	-	-	-	-	-	-	-

^a Standard deviation; ^b Bottom stream.

The behavior of the top product concentration during distillation is shown in Figure 1.a. It is observed that the distillation of solution number 6 was more stable in terms of the top product concentration (lowest standard deviation – see Table 3). The stability of the operation with the solution 6 is corroborated by a lower standard deviation observed for the reflux ratio. Other experiments demonstrated that distillation required a larger time interval for the process to reach steady state. This delay can most likely be attributed to the stabilization of compositions along the distiller. As discussed by Webb and Ingraham (1963), the water forms azeotropes with alcohols in all experiments analyzed for a range of water concentrations from 4.0 to 49.6 % m/m.

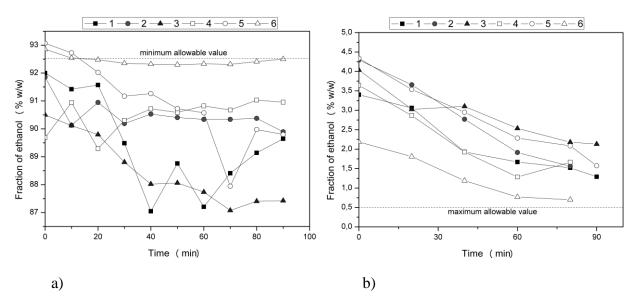


Figure 1. Continuous distillation behavior of: a) top product concentration, and b) bottom product concentration.

These results show that limiting the presence of fusel oil in HEF would depend only on its influence on engine performance. However, ANP Technical Notes 04- and 97-/2013/SBQ/CPT-101-DF states that fusel oil do not affect the performance of combustion engines. In fact, the fusel oil affect positively the energy contained in the HEF because its higher enthalpy of combustion,



e.g., the enthalpy of combustion of 3-methyl-1-butanol is approximately 30% higher than that of ethanol.

4. CONCLUSION

The results confirmed that the presence of higher alcohols during the distillation of ethanol may influence the concentration of both the top and bottom products. However, these alcohols do not prevent the attainment of HEF with ethanol concentration as required by legislation. The main challenge raised by these experiments is the high loss of ethanol in the bottom product. The collected information suggests that this problem may arise from the insufficient number of separation stages in stripping section. The influence of the fusel oil components has little effect because the reference mixture (ethanol + water) showed a similar behavior. It is important to note that the higher alcohol concentrations in the feed stream were overestimated, thus the influence of these components on the distillation process tends to be lower than observed.

From the data obtained, it can be stated that there is no need to remove fusel oil during the distillation in small-scale fuel ethanol production. Eliminating this process would lower investment and operating costs. This statement is supported by the fact that legislation regulating the commercialization of HEF does not impose limits on the content of higher alcohols. Thus, limiting the presence of fusel oil in HEF only benefits engine performance. The constructive and operational simplification arising from these results can aid in the technical and economic feasibility of ethanol production on a small scale.

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