Ethanol dehydration via azeotropic distillation with gasoline fraction mixtures as entrainers: A pilot-scale study with industrially produced bioethanol and naphtha.

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Abstract

Various hydrocarbons (n-hexane, cyclohexane, toluene, isoctane) and mixtures of them (binary, ternary or quaternary), as well as two different types of industrially produced naphtha (one obtained by direct distillation and the other from a catalytic cracking process), have been tested as candidate entrainers to dehydrate ethanol. The tests were carried out in an azeotropic distillation column on a semi pilot plant. The results show that it is possible to dehydrate bioethanol using naphtha as entrainer, obtaining as a result a fuel blend with negligible water content and ready for immediate use in motor vehicles.

1. Introduction

In order to reduce the greenhouse gas emissions produced by the transportation sector, the use of biomass as a fuel or in a mixture with fossil fuels has been promoted by government legislative and financial initiatives. Among the various biofuel technologies, bioethanol has advanced the most. However, bioethanol obtained from the fermentation of biomass, which can come from different sources, needs to undergo several steps of purification. This bioethanol is produced in an aqueous media and must thus be dehydrated before it can be used as a fuel. European legislation permits a maximum of 0.3% water by weight fraction in the bioethanol to be blended with gasoline [1].

Even though the presence of a binary azeotrope in the water + ethanol mixture makes it impossible to separate ethanol and water in a single distillation step, there exists many other techniques [2] that can be used, alone or in combination, to dehydrate ethanol. Some of them include, but are not limited to: adsorption on molecular sieves [3], azeotropic distillation [4], pressure swing distillation [5], pervaporation [6], extractive distillation with ionic liquids [7], etc.

Concretely, in recent years several papers have treated bioethanol dehydration through only simulation testing different techniques such as pressure swing absorption [8], hybrid processes (distillation/adsorption/vapor permeation [9], based on liquid-liquid extraction [10], heteroazeotropic distillation using a gasoline additive as entrainer [11], extractive batch distillation [12] or a heat-pump-assisted extractive distillation in a single step [13] and comparing them to the conventional process. Though all the techniques presented in these papers seem viable alternatives and arouse improvements in bioethanol dehydration process, the results presented are almost all based on

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simulation and lack in the majority of cases experimental validation. Thus it is necessary experimental data regarding non-conventional processes for ethanol dehydration.

These techniques are more or less energy demanding depending on the technique and the separation requirements. Traditionally, the ethanol is purified completely before being used in a fuel blend. However, in the novel process proposed in a previous paper [14], based on heterogeneous azeotropic distillation, the immediate product already is a fuel blend containing little water, meaning that it can be used directly or added to another fuel on the contrary of the other processes proposed in papers where dehydrated ethanol has to be mixed afterwards and thus intensifying the whole process.

In a previous study, several pure hydrocarbons (hexane, cyclohexane, toluene and isooctane) [14] were tested as entrainers in the ethanol dehydration process. The results showed that it was possible to obtain a hydrocarbon + ethanol mixture with a negligible water content. The continuation of that research is the subject of the present article: a study of the possibility of using hydrocarbon mixtures as entrainers in the industrial bioethanol dehydration process, with a view to assessing the viability of a process where a gasoline and ethanol mixture containing a negligible amount of water can be obtained directly.

The research has been carried out in two stages:

1. Several binary and ternary hydrocarbon mixtures, combinations of those investigated previously, [14] were tested as entrainers to verify their suitability for the ethanol dehydration process.

2. Two different types of naphtha, obtained from a refinery, were tested in combination with industrial bioethanol in order to investigate the possibility of using a more complex mixture as entrainer and so, by the above process, directly manufacture a complex fuel blend.

2. Experimental procedure

2.1. Chemicals

For the first stage of the study, analytical grade hydrocarbons supplied by PANREAC AppliChem (ITW reagents) provider were used. Their purities were as follows: hexane (min 98.5%), cyclohexane (min 99.5%), isooctane (min 99%) and toluene (min 99%) as stated by provider. The analytical grade ethanol and 2-propanol used to prepare the standards were supplied by MERCK provider, (with purities min 99.9 and 99.8%, respectively as stated by provider). The deionized water that was used had a measured conductivity of 3 µS/cm.

For the second stage, bioethanol produced in a single distillation step on an actual fermentation plant, was used directly (Abengoa Plant, Valle de Escombreras-Cartagena (Spain)). Bioethanol water content was checked against the Karl-Fischer (Mettler-Toledo model DL-31) technique and was, on average, 8.3% by weight fraction. The degree of purity of the bioethanol was measured in a gas chromatograph (Agilent 7890B) coupled to a high resolution mass spectrometer (Agilent 7200(Q-TOF)). It was found to contain 1-propanol, isobutanol and ethyl acetate in quantities less than 1% by mass each. Other compounds were also detected during the analysis, but the sum total
of their concentrations amounted to less than 1%.

The bioethanol was analyzed in an Ionized Coupled Plasma detector coupled to a mass chromatograph (Perkin Elmer, Model: 7300 DV (with dual vision)) to determine its mineral content in S, P and Cu. Furthermore, ionic chromatography (Metrohm 850 ProfIC AnCat- MCS) was used to analyze for SO₄ and Cl and determine their concentrations. The results of the analyses are presented in Table 1.

As can be seen, the inorganic content of the bioethanol is far below legal limits. The limits quoted in Table 1, as per the European regulation EN 15376:2011 [1], refer to ethanol that is directly mixed with fuel. In the case of the present study, the ethanol is dehydrated during the process itself. As a result, the limit for inorganics should correspond to that of the final fuel. That is, if there can be at most 10% ethanol in the final fuel then the concentration of the inorganics in the bioethanol + fuel mixture should not exceed a limit of 10% in the dehydrated ethanol.

The two types of naphtha that were used were provided by Repsol (Cartagena plant - Spain). Naphtha 1 was obtained by direct distillation of petrol whereas naphtha 2 was obtained by the catalytic cracking of higher boiling point petrol fractions. The characteristics of both are recorded in Table 2 and Table 3. These characteristics are quoted by the provider and were checked in this work by means of a 1:100 split-injection mass chromatography analysis on both naphtha types, during which a temperature ramp of 5°C/min was applied, following the procedure outlined in regulation ISO 22854:2014 [15]. A high resolution mass spectrometer (Agilent 7200(Q-TOF)) coupled to a gas chromatograph (Agilent 7890B) was employed to this end. As can be seen in Table 3, naphtha 2 has a higher than usual content in olefins and aromatics as would be expected from the cracking process.

The distillate curves of the two naphtha types, presented in Figure 1a, have been obtained in keeping with regulation ISO 3405:2011 [16]. Figure 1b shows the chromatographs of both naphtha types. Their volatilities are fairly similar even though naphtha 1 has a slightly wider temperature range between the initial and final evaporation points than naphtha 2, as can be appreciated from Figure 1a.

### 2.2. Procedure

The first part of the study followed the procedure described in a previous paper ([14]). A 50 mm diameter Armfield UOP3CC column built to the scale of a semi pilot plant, and fitted with eight plates and covered with Armaflex AF (Armacell Advanced Insulation) insulating material, was used to this end. Two preheated feeds were let onto the first plate, while the heating power in the boiling chamber was varied. A decanter with two outlets after the condenser permitted one of the condensed vapor phases to return to the column.

The flow diagram of the process is presented in Figure 2. The two feeds are pumped inside the column thanks to two peristaltic pumps. The hydrocarbon mixture goes through a heat exchanger (HE-6) where it is preheated with the bottom product of the column. Later on this feed and the ethanol one go through two heat exchangers where they are preheated thanks to an oil heating flux (HE-4 and HE-5). Those two preheated feeds enter the column at the top stage. The vapor exiting the top stage of the column
condensates inside the condenser (HE-2) where cold water acts as a refrigerant. The condensate arrives to a decanter where it splits in two phases. The organic phase returns to the column after a heating step (HE-3) in the first stage. The aqueous phase containing the majority of water is discharged. The bottom product is the desired product the ethanol + hydrocarbon mixture with little water content and exits from the boiling chamber (HE-1) at half of the boiling chamber height. The TM symbols in the figure correspond to thermopar sensors connected to the main control equipment for monitoring and saving the temperature data all along the process.

In Table 4 are presented the experimental conditions (flow rates, compositions, temperatures…) for each of the experiments on the different mixtures.

The feed flow rate was maintained constant between experiments. The organic phase from the decanter was returned to the column while the aqueous phase was treated separately. The flow rates of ethanol and entrainer presented in Table 4 permits the obtaining of a fuel blend with an ethanol content of up to 10% by mass (objective for biofuel in transport fuel in the EU for the year 2020)[17].

The samples collected from the distillate and bottom product were analyzed by gas chromatography in a Shimadzu GC-14B gas chromatograph with a PORAPACK Q column coupled to a Thermal Conductivity Detector (TCD). The carrier gas was helium at a flow rate of 50 mL/min and an oven temperature of 170 ºC. As the obtained samples were very complex (many different hydrocarbons coming from the naphtha feed), only the ethanol and water content were quantified. The water content was checked against the Karl Fischer technique.

3. Experimental results

3.1. Hydrocarbon mixtures

Figures 3 through 6 are plots of different variables against the power used in the boiling chamber, and correspond to the experimental data that have been obtained. Figures 3a, 4a, 5a and 6a show the flow rates of the distillate and bottom product. Figures 3b, 4b, 5b and 6b show the hydrocarbon and water content in the distillate. Figures 3c, 4c, 5c and 6c show the hydrocarbon composition of the bottom product. Finally, figures 3d, 4d, 5d and 6d show the water content in the bottom product.

As can be deduced from the figures, there exists a heating power for which it is possible to obtain a fuel blend containing less than 100 ppm water, accompanied by a variation in ethanol content of between 5 and 10%.

When the power is increased, the ethanol content in the bottom product tends to fall as the water content does, except for the experiments with mixtures containing hexane. In these two cases (Figures 5c and 6c) it is observed that the ethanol content remains approximately constant in the power range tested. An optimal power appears to result in a maximum ethanol concentration in the bottom product, corresponding to a water content that is below the legal limit. Obtaining a fuel blend where the water/ethanol mass content ratio is 0.003 would be an optimum situation. To achieve this situation, for instance, in the experiments with the hexane-cyclohexane-isoctane a power of around
90 W is required. Based on the flow rates used and experimental conditions (preheating step, heat loses...) 1.7 kJ/g ethanol or 0.47 kWh/kg ethanol dehydrated is required. While this energy cost is low compared to conventional process (e.g. 2.07 kWh/kg [13]) it takes only into account the experimental heat duty in the boiling chamber. This step should be integrated in the heat exchanger network of a refinery to analyze its industrial viability.

The hydrocarbon content in the bottom product increases as the power does (except for experiment 3 and 4 where the hydrocarbons/ethanol proportion remains almost constant as stated before). An increase in the hydrocarbon content of the distillate is also observed in all of the experiments. This indicates somewhat of a loss of volatile compounds in the distillate as the power increases. It also highlights the existence of an optimal power for the process that avoids hydrocarbon losses in the distillate. For instance, for the optimal power shown previously the ethanol recovery rate in the bottom product is 78.4%, while for the hexane, cyclohexane and isooctane is 97.5, 98.4 and 99.4% respectively.

Moreover, while it is not shown in the figures, the existence of a maximum heating power beyond which the desired separation no longer takes place, has been observed. In fact, when the power is too high the vapor obtained in the distillate becomes homogeneous and thus the decanter cannot separate an aqueous rich fraction from the column.

### 3.2. Naphtha feed

In figures 7 and 8, the same variables as in the experiments on the hydrocarbon mixtures are plotted, again as a function of the power used in the boiling chamber. However, due to the complexity of the mixtures, in this case only ethanol and water content have been plotted.

As in the earlier case, it can be seen that an increase in the power employed increases the distillate flow rate and reduces that of the bottom product. More power reduces the water content in the bottom product to below the detection limit of 50 ppm.

On the other hand, the greater the power used the more ethanol and naphtha compounds appear in the distillate. As a result, an optimum power exists for which the water content in the bottom product is below the limit and the flow rates of the ethanol and naphtha compounds in the distillate are minimized, avoiding in this case the loss of valuable compounds. Nevertheless, subjecting the distillate to another separation step might make it possible to recover of some of those compounds.

### 5. Conclusions

In this paper, on the one hand, it has been studied the performance of four hydrocarbons (n-hexane, cyclohexane, toluene, isooctane) and their binary, ternary and quaternary mixtures to dehydrate ethanol obtaining a fuel blend. On the other hand, the performance of two different types of industrially produced naphtha (one obtained by direct distillation and the other from a catalytic cracking process) has been checked.
Experiments under similar conditions have been performed in an azeotropic distillation column on a semi pilot plant scale to compare the technical viability of the hydrocarbons to dehydrate ethanol. In these experiments, the flow rates and the composition has been analyzed with the power increase. It has been observed that in most of the experiments carried out, there is a maximum heating power beyond which the desired separation no longer takes place.

It has been proved that the use of a heterogeneous azeotropic process to dehydrate bioethanol and directly obtain a fuel blend is possible for many hydrocarbon mixtures, including complex ones such as naphtha.

The next step in the research would be to analyze the costs (material and energy) of the proposed process and then to integrate it into a refinery/biofuel production plant in order to verify to what extent the dehydration of ethanol is improved relative to conventional processes.

References:


Table 1. Inorganic Content of bioethanol

<table>
<thead>
<tr>
<th>Compound</th>
<th>Content (ppm)</th>
<th>Legislation limit (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>&lt;3</td>
<td>10</td>
</tr>
<tr>
<td>SO₄</td>
<td>Not detected</td>
<td>4.0</td>
</tr>
<tr>
<td>P</td>
<td>No detected</td>
<td>0.15</td>
</tr>
<tr>
<td>Cu</td>
<td>0.023</td>
<td>0.1</td>
</tr>
<tr>
<td>Cl</td>
<td>0.25</td>
<td>6.0</td>
</tr>
<tr>
<td>NO₃⁻</td>
<td>0.28</td>
<td>No limit</td>
</tr>
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</table>

Table 2. Composition of the two naphtha types, in mass percent, as a function of their carbon number in the molecules.

<table>
<thead>
<tr>
<th>%w/w</th>
<th>naphtha 1</th>
<th>naphtha 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>C5</td>
<td>0</td>
<td>0.068</td>
</tr>
<tr>
<td>C6</td>
<td>8.712</td>
<td>3.805</td>
</tr>
<tr>
<td>C7</td>
<td>33.502</td>
<td>33.936</td>
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<tr>
<td>C8</td>
<td>37.603</td>
<td>52.415</td>
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<tr>
<td>C9</td>
<td>18.405</td>
<td>9.236</td>
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<tr>
<td>C10</td>
<td>1.653</td>
<td>0.503</td>
</tr>
<tr>
<td>C11</td>
<td>0.096</td>
<td>0</td>
</tr>
<tr>
<td>C12</td>
<td>0</td>
<td>0.022</td>
</tr>
</tbody>
</table>
Table 3. PIONA (w/w%) analysis of the two naphtha types

<table>
<thead>
<tr>
<th>Type of hydrocarbon</th>
<th>naphtha 1</th>
<th>naphtha 2</th>
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<tbody>
<tr>
<td>p6</td>
<td>3.916</td>
<td>0.670</td>
</tr>
<tr>
<td>p7</td>
<td>18.369</td>
<td>4.547</td>
</tr>
<tr>
<td>p8</td>
<td>18.385</td>
<td>11.459</td>
</tr>
<tr>
<td>p9</td>
<td>10.674</td>
<td>2.981</td>
</tr>
<tr>
<td>p10</td>
<td>1.288</td>
<td>0.503</td>
</tr>
<tr>
<td>p11</td>
<td>0.096</td>
<td>0.000</td>
</tr>
<tr>
<td>N5</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>n6</td>
<td>4.155</td>
<td>0.482</td>
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<tr>
<td>n7</td>
<td>10.745</td>
<td>5.157</td>
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<tr>
<td>n8</td>
<td>11.877</td>
<td>6.007</td>
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<tr>
<td>n9</td>
<td>6.772</td>
<td>1.834</td>
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<tr>
<td>n10</td>
<td>0.226</td>
<td>0.000</td>
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<tr>
<td>n11</td>
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<td>0.000</td>
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<tr>
<td>a6</td>
<td>0.641</td>
<td>0.105</td>
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<tr>
<td>a7</td>
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<td>9.277</td>
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<td>a8</td>
<td>7.341</td>
<td>21.562</td>
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<tr>
<td>a9</td>
<td>0.959</td>
<td>2.395</td>
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<tr>
<td>a10</td>
<td>0.139</td>
<td>0.000</td>
</tr>
<tr>
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<tr>
<td>O6</td>
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<td>O7</td>
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<tr>
<td>O8</td>
<td>-</td>
<td>13.386</td>
</tr>
<tr>
<td>O9</td>
<td>-</td>
<td>2.025</td>
</tr>
<tr>
<td>O10</td>
<td>-</td>
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<table>
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<tr>
<th></th>
<th>P%</th>
<th>N%</th>
<th>A%</th>
<th>O%</th>
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<tbody>
<tr>
<td>naphtha 1</td>
<td>52.728</td>
<td>33.775</td>
<td>13.468</td>
<td>-</td>
</tr>
<tr>
<td>naphtha 2</td>
<td>20.16</td>
<td>13.48</td>
<td>33.34</td>
<td>33.02</td>
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<table>
<thead>
<tr>
<th></th>
<th>% n-parafins</th>
<th>% isoparafins</th>
<th>% n-olefins</th>
<th>% iso-olefins</th>
<th>% naphtheno-olefins</th>
<th>% dio-olefins</th>
<th>% Other olefins</th>
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<tr>
<td>naphtha 1</td>
<td>17.83</td>
<td>34.90</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>naphtha 2</td>
<td>2.27</td>
<td>17.89</td>
<td>6.3</td>
<td>12.22</td>
<td>12.84</td>
<td>1.21</td>
<td>0.45</td>
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Table 4: Experimental conditions in each of the experiments

<table>
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<tr>
<th>Experiment</th>
<th>Feed 1</th>
<th>Feed 2</th>
<th>Flow rate Feed 1 g/min</th>
<th>Flow Rate Feed 2 g/min</th>
<th>Water %w/w in Feed2</th>
<th>T (°C) Feed Stage</th>
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</thead>
<tbody>
<tr>
<td>1. Binary entrainer</td>
<td>50% w/w Hexane; 50% w/w Isooctane</td>
<td>Ethanol with water</td>
<td>41.6</td>
<td>4.34</td>
<td>6</td>
<td>62</td>
</tr>
<tr>
<td>2. Binary entrainer</td>
<td>50% w/w Cyclohexane, 50% w/w Isooctane</td>
<td>Ethanol with water</td>
<td>41.63</td>
<td>4.38</td>
<td>10</td>
<td>65</td>
</tr>
<tr>
<td>3. Ternary entrainer</td>
<td>33% w/w Hexane, 33% w/w Cyclohexane, 33% w/w Isooctane</td>
<td>Ethanol with water</td>
<td>41.38</td>
<td>4.4</td>
<td>7</td>
<td>62</td>
</tr>
<tr>
<td>4. Quaternary entrainer</td>
<td>25% w/w Hexane, 25% w/w Cyclohexane, 25% w/w Isooctane, 25% w/w Toluene</td>
<td>Ethanol with water</td>
<td>42.34</td>
<td>4.34</td>
<td>6</td>
<td>68</td>
</tr>
<tr>
<td>5. Complex mixture</td>
<td>Naphtha 1 from direct distillation</td>
<td>Bioethanol</td>
<td>43</td>
<td>4.3</td>
<td>8.3</td>
<td>63</td>
</tr>
<tr>
<td>6. Complex mixture</td>
<td>Naphtha 2 from FCC process</td>
<td>Bioethanol</td>
<td>44.5</td>
<td>4.3</td>
<td>8.3</td>
<td>63</td>
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</table>
Figure 1a. Distillation curves of the two types of naphtha studied.
Figure 1b. Chromatographs with mass analysis for naphtha 1 (above) and naphtha 2 (below).
Figure 2. Experimental flow diagram of the tested process
Figure 3. Mixture containing hexane and isoctane. Plot of several variables against reboiler heat duty: a) Experimental top (♦) and bottom (■) product flow rates. b) Experimental composition (w/w) of water, ethanol hexane and isoctane in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, hexane and isoctane in the column bottom. d) Experimental composition (w/w) of water in the bottom product.
c) 0,0002
0,0004
0,0006
0,0008
0,001
0,0012
0 50 100 150 200 250 300 350 400
Power (W)

0,0012
0,001
0,0008
0,0006
0,0004
0,0002
0
0 50 100 150 200 250 300 350 400
Power (W)

Ethanol
Hexane
Isooctane
Figure 4. Mixture containing cyclohexane and isooctane. Plot of several variables against reboiler heat duty: a) Experimental top (♦) and bottom (■) product flow rates. b) Experimental composition (w/w) of water, ethanol cyclohexane and isooctane in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, cyclohexane and isooctane in the column bottom. d) Experimental composition (w/w) of water in the bottom product.
b) Weigth Fraction vs Power (W)

- Water
- Ethanol
- Ciclohexane
- Isooctane

c) Weigth Fraction vs Power (W)

- Ethanol
- Cyclohexane
- Isooctane
d)
Figure 5. Mixture containing hexane, cyclohexane and isooctane. Plot of several variables against reboiler heat duty: a) Experimental top (♦) and bottom (■) product flow rates. b) Experimental composition (w/w) of water, ethanol, hexane, cyclohexane and isooctane in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, hexane, cyclohexane and isooctane in the column bottom. d) Experimental composition (w/w) of water in the bottom product.
Weigth Fraction

Power (W)

- Ethanol
- Hexane
- Cyclohexane
- Isooctane

0 20 40 60 80 100 120 140 160

Weigth Fraction

Power (W)
Figure 6. Mixture containing hexane, cyclohexane, isooctane and toluene. Plot of several variables against reboiler heat duty. a) Experimental top (♦) and bottom (■) product flow rates. b) Experimental composition (w/w) of water, ethanol, hexane, cyclohexane, isooctane and toluene in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, hexane, cyclohexane, isooctane and toluene in the column bottom. d) Experimental composition (w/w) of water in the bottom product.
c) 

![Graph showing weight fraction vs. power (W)](image_url)

- Ethanol
- Hexane
- Cyclohexane
- Isooctane
- Toluene

0,05
0,1
0,15
0,2
0,25
0,3

0 50 100 150 200 250 300

Power (W)

0

0,0001
0,0002
0,0003
0,0004
0,0005
0,0006
0,0007

0 50 100 150 200 250 300

Power (W)

Weight Fraction

0,0001
0,0002
0,0003
0,0004
0,0005
0,0006
0,0007
Figure 7. Naphtha 1 Mixture. Plot of several variables against reboiler heat duty: a) Experimental top (♦) and bottom (■) product flow rates. b) Experimental composition (w/w) of water (♦) and ethanol (■), in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, in the column bottom. d) Experimental composition (w/w) of water in the bottom product.
c) 

\begin{figure}
\centering
\includegraphics[width=\textwidth]{image_c}
\caption{Weight Fraction vs Power (W) for (a) and (b).}
\end{figure}

d) 

\begin{figure}
\centering
\includegraphics[width=\textwidth]{image_d}
\caption{Weight Fraction vs Power (W) for (c) and (d).}
\end{figure}
Figure 8. Naphtha 2 Mixture. Plot of several variables against reboiler heat duty: a) Experimental top (◊) and bottom (■) product flow rates. b) Experimental composition (w/w) of water (◊) and ethanol (■), in the column distillate (aqueous phase). c) Experimental composition (w/w) of ethanol, in the column bottom. d) Experimental composition (w/w) of water in the bottom product.