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Ethanol dehydration via azeotropic distillation with gasoline fractions as entrainers: A pilot-scale study of the manufacture of an ethanol-hydrocarbon fuel blend.

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Abstract

We establish experimentally and through simulations the economic and technical viability of dehydrating ethanol by means of azeotropic distillation, using a hydrocarbon as entrainer. The purpose of this is to manufacture a ready-to-use ethanol-hydrocarbon fuel blend. In order to demonstrate the feasibility of this proposition, we have tested an azeotropic water-ethanol feed mixture, using a hydrocarbon as entrainer, in a semi pilot-plant scale distillation column. Four different hydrocarbons (hexane, cyclohexane, isooctane, and toluene) that are representative of the hydrocarbons present in ordinary gasoline have been tested. Each of these hydrocarbons was tested separately in experiments under conditions of constant feed rate and variable reboiler heat duty. The experimentally obtained results are compared with results calculated by a simulator. Finally, the proposed and traditional ethanol dehydration processes are compared to ascertain the advantages of the former over the latter.

1. Introduction

The fermentation of biomass represents a promising means of obtaining biofuels – one that involves lower emissions of polluting gases into the atmosphere. Bioethanol is one of the most widely used of these biofuels, and the manufacture thereof from raw materials requires several rounds of treatment, including milling, fermentation and distillation, before a water-ethanol azeotrope can be obtained. A dehydration step usually follows at this point, for which there are many different available techniques [1, 2], such as adsorption on molecular sieves [3], azeotropic distillation [4], pressure swing distillation [5], pervaporation [6], extractive distillation with ionic liquids [7], etc.

Traditional methods of bioethanol manufacture involve producing pure ethanol for subsequent addition to a fuel blend. In the past, azeotropic distillation was the most commonly used method to obtain pure ethanol using benzene [8], ciclohexane [9], hexane [10], toluene [11], isooctane [12]… as entrainers. Currently the bioethanol is generally dehydrated by the biofuel manufacturer by means of molecular sieves, and then kept in storage for later addition to gasoline in legally mandated proportions. At present, European legislation mandates 10% [13] as the maximum permitted gasoline ethanol-content, and 0.3% [14] as the maximum permitted bioethanol water-content.

However, the novel process that we present in this paper would lead to the manufacture of an ethanol-hydrocarbon blend that is immediately ready for use as fuel, with gasoline itself serving as the separating agent. In practice, this blend would be manufactured at refineries via azeotropic distillation,

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and then distributed directly to gas stations.

Gasoline is a complex mixture of hydrocarbons and our intention here is to embark on a systematic study of the process that is the subject of this paper. So, as the first step in a more ambitious research effort, we have selected a series of hydrocarbons we consider to be representative of the different hydrocarbon fractions present in gasoline. Thus, to represent the straight-chain hydrocarbon fractions in gasoline, we chose hexane; to represent cyclic hydrocarbons, we chose cyclohexane; isooctane for branched hydrocarbons; finally, toluene served to represent the aromatic fractions. Each of these was tested in an azeotropic distillation column at the semi pilot-plant scale, in order to gauge the technical feasibility of a process that obtains such a ready-to-use fuel blend. To establish whether the hydrocarbons are suitable for use in this process under different operating conditions, as well as for purposes of comparison with the conventional process, we have simulated the experiments using equilibrium data.

Ultimately, our intention is to show that it is both technically and economically feasible to obtain, via azeotropic distillation, a bioethanol-hydrocarbon fuel blend that is immediately ready for use in modern motor vehicles. Additionally, the new process would represent an improvement over current best practice.

2. Materials and methods

2.1Chemicals

The chemical purity grades of the tested hydrocarbons were as follows: hexane (min 98.5%), cyclohexane (min 99.5%), isooctane (min 99%) and toluene (min 99%); all were purchased from the commercial supplier PANREAC. Analytical grade ethanol and 2-propanol, supplied by MERCK, were used to prepare the standards (min 99.9 and 99.8%, respectively). The deionized water that was used had a measured conductivity of 3 µS/cm.

2.2 Equipment

A 50mm diameter Armfield UOP3CC column, fitted with eight plates and built to the scale of a semi pilot plant was used. Details of the equipment can be found in a prior publication [15]. Only one minor modification was made to it for the purpose of the present study: the column and the boiling chamber were wrapped with thermal insulation. The insulating material was Armaflex AF (Armacell Advanced Insulation), supplied by PecoMark. Figure 1 shows a schematic diagram of the experimental setup. The label C stands for column, D is for decanter, HE denotes various heat exchangers and TM refers to the many points where sampling took place.

Based on the capacity and characteristics of the column, as well as our objective of obtaining a 5% w/w ethanol concentration in the bottoms product, below the legal limit, the flow rate of the various hydrocarbon entrainers was fixed at 41 g/min, while the water-ethanol mixture's was 4.38 g/min. The composition of the latter was approximately 93% w/w ethanol and 7% w/w water, which is close to composition of the binary azeotrope. The temperature of the various streams and process units varied depending on the hydrocarbon, and took into account its boiling point temperature. Table 1 lists those temperatures:

2.3 Sampling and analysis

The compositions of the samples were determined by chromatography with 2-propanol as internal standard at a standard deviation of 2%. The composition of the water was compared with that obtained

by Karl-Fischer titration at a standard deviation of 1%. A detailed description of the sampling and analytical methods used to determine the concentrations of the column exit streams can be found elsewhere[15].

3. Experimental Results

Multiple experiments were carried out on each of the hydrocarbons. There are many variables that can affect the distillation process: the mass ratio between ethanol and entrainer, the reboiler heat duty, the entrainer compound, flow rates, temperatures, etc. Only two of these variables were chosen for studying the process, the entrainer and heat duty, while the rest were kept constant. Notice that the purpose of this study was to ascertain the feasibility of the proposed process as well as to identify problems that might arise in the industrial implementation thereof.

Once steady state had been reached (after at least an hour), which was ascertained by repeatedly measuring the water content in the bottoms stream, we also measured flow rates and determined the compositions of all streams. With this information, it is possible to verify whether the conditions of material balance had been satisfied at all the sampling points. Of all the concentrations tested, the bottoms stream water-content was given particular attention since excess water in the fuel blend would mean it no longer met legal requirements.

Figures 2 to 5 show three graphs of several results obtained for each tested hydrocarbon, plotted as a function of reboiler heat duty. Each Figure contains a plot of the flow rates of the top and bottoms streams of the column (Figures 2-5 a), the ethanol and hydrocarbon concentrations in the bottoms (Figures 2-5 b) and finally, the water concentration in the bottoms (Figures 2-5c). It is the latter quantity that permits assessing the feasibility of the process.

It was found in all cases that there was a maximum value of the reboiler heat duty beyond which the column no longer operated correctly. This was evident from the fact that the vapor leaving the top of the column did not split into two liquid phases on cooling. However, we must emphasize that neither this maximum nor any of the other reboiler heat duties can be compared across different mixtures since they depend on other operating variables such as the temperature of the hydrocarbon in the column feed; this differs from hydrocarbon to hydrocarbon simply because they have different boiling point temperatures, as shown in Table 1.

Figures 2-5 a show that the distillate flow rates are small compared with the overall feed rate (about 45 g/min). They remain essentially constant or increase slightly with the exception of the system containing cyclohexane, whose flow rate increases markedly.

As regards the bottoms stream flow rates (Figures 2-5 a), they are roughly the same as the feed flow rate (45 g/min), and as in the case of the distillate, also remain essentially constant with the exception of the mixture containing cyclohexane (Figure 3 a), which experiences a marked decrease in flow rate as the reboiler heat duty increases.

The mass fraction of water in the waste stream (Figures 2-5 c) is the key parameter here since it will allow us to determine whether it is actually feasible to produce a fuel blend by ethanol dehydration using the method presented in this paper. In this regard, we observe the same behavior of all the mixtures we tested: on increasing the reboiler heat duty, the mass fraction of water in the waste stream decreases sharply. This is how we achieved our objective of obtaining an ethanol-hydrocarbon product that is nearly water-free: by increasing the reboiler heat duty. After the period of rapid decrease in the mass fraction of water, which remained very low subsequently, we observed only small fluctuations in its value. In any event, the obtained values did not exceed the maximum legal limit of 0.00015 (% w/w), which is equivalent to a maximum of 0.3% ethanol water-content in a blend containing 5% ethanol[14].

On the other hand, the mixture containing cyclohexane (Figure 3) behaved rather differently. The observed fluctuations in flow rates and compositions of this mixture are significantly larger than in the case of the other mixtures. This likely has to do with the small differences in shape of the solubility curve between this mixture and the others in the working range of the distillation column. This working range is close to the mixture's plait point, and there usually is not much experimental liquid-liquid equilibrium data available in this range to permit a detailed study. Such data are not easy to determine since small changes in the composition of the global mixture lead to large ones in the composition of the equilibrium liquid phases.

In conclusion, it is important to point out that all the studied hydrocarbons are suitable for producing an ethanol-hydrocarbon mixture that is nearly free of water. Consequently, it is not unreasonable to expect this to apply also to a mixture of several hydrocarbons, which would permit the eventual use of gasoline as entrainer in the manufacture of a ready to use ethanol-hydrocarbon fuel blend.

4. Simulation

For a proper design of the ethanol dehydration process considered here, commercial simulation software packages must possess a series of equations and parameters that will enable calculation of both the vapor-liquid equilibrium in the distillation column as well as the liquid-liquid and vapor-liquid-liquid equilibria in the decanter. In general, thermodynamic models tend to reproduce these equilibria more or less correctly. However, as was pointed out in another study [16], near the plait

point in the heterogeneous region of the mixture there always arises a systematic discrepancy between experimental and thermodynamically modeled data. The models generally produce a heterogeneous region for water-ethanol-hydrocarbon mixtures that is larger than is observed experimentally, and especially near the plait point, which happens to overlap with the working range of the decanter, as mentioned earlier. Furthermore, these models are known to fail to reproduce the marked asymmetry observed in the non-isothermal binodal curves of the above mixtures. As a general rule, the heterogeneous regions calculated from correlation parameters are always larger and shifted more toward the ethanol-hydrocarbon binary than is observed experimentally [17, 18, 19, 20].

The consequence is that errors arise when trying to simulate the proposed process by means of commercial software employing conventional thermodynamic models to determine the liquid-liquid equilibrium. The most glaring error is a failure to predict a maximum reboiler heat duty at which point the process ceases to run correctly, when experimentally this is exactly what is observed because two phases no longer appear in the decanter. This error arises because the size of the heterogeneous region calculated by the model is larger than is found experimentally, and is, as was pointed out earlier, exactly what thermodynamic models would predict for water-ethanol-hydrocarbon mixtures.

The various experiments conducted on each of the hydrocarbon mixtures in the azeotropic distillation column have been simulated by means of the commercially available software Chemcad 6 [21]. The operating variables were assigned the same values as in the laboratory experiments. The UNIFAC model was used to determine the vapor-liquid equilibrium data because the equilibrium data calculated in this way have been found to compare favorably with experiment. However, it was necessary to design a special process unit to calculate the separation of the liquid phases in the decanter. In an attempt to avoid the discrepancies encountered in correlations using conventional models, the liquid-liquid equilibrium data were instead calculated by means of interpolation of experimental data from the literature. This was accomplished by making simultaneous use of two equations: on the one hand, a “spline” interpolation [22] was used, which is a set of cubic interpolations through a minimum of five points, to fix the position of the binodal curve; on the other, the ratio between the equilibrium aqueous and organic phases was determined via Hand’s equation [23].

Figure 6 shows the simulation scheme, and includes the following process units:

Unit 1 (C): Distillation column. The column used in the experiments was fitted with 8 plates and a reboiler. The efficiency of the distillation stages in simulations has been estimated to be 0.6-0.7 following the criterion by Stichlmair y Fair 1998 [24]. Therefore, based on this, we selected a column with 6 equilibrium stages. The column does not use a condenser, whose function is instead carried out by unit 2 in Figure 6.

Unit 2 (HE-1): Heat exchanger. This unit controls the temperature of vapor stream 8, which leaves from the top of the column. This temperature is set to the temperature at which decanting of phases takes place in the experiments.

Unit 3 (U-1): This unit determines whether stream 9 contains one or two liquid phases. If the composition of stream 9 is in the homogeneous region (according to the curve obtained by the “spline” interpolation), it is recycled back to the column. If it falls in the heterogeneous region, on the other hand, the stream passes on to unit 4.

Unit 4 (U-2): This process unit carries out the separation of phases by taking into account both the spline interpolation and the correlation via Hand’s equation. The phase of largest hydrocarbon mass

fraction leaves in stream 4 and is recycled back to the column, while the one containing the most water leaves in stream 5.

Unit 5 (M): This combines streams 4 and 11. Note that unit 5 receives either one or both of these streams, which is determined by unit 3.

Unit 6 (HE-2): This heat exchanger raises the temperature of the recycle stream to the experimental working temperature.

Based on the flow diagram in Figure 6 and by varying only the supplied reboiler heat duty, we have simulated the experiments. The results are plotted in Figures 2 to 5, which also show the experimental results. Since heat losses to the surroundings are not accounted for in the simulations, the simulation reboiler heat duty values are shifted with respect to the experimental values on the horizontal axes.

First of all, it is worth mentioning that the above method of calculating the liquid-liquid equilibrium produces a maximum reboiler heat duty beyond which azeotropic distillation is no longer possible. This result is not obtained when models such as NRTL, UNIQUAC or UNIFAC are used to determine the liquid-liquid equilibrium, and is a consequence of the prediction by these models, with rising reboiler heat duty, of a composition of the vapor leaving the top of the column that lies in the heterogeneous region, when in reality it should lie in the homogeneous region.

On comparing the simulation and experimental results, we observe that very similar bottoms and top product flow rates are obtained, respectively. The greatest deviation from this trend occurs in the case of the mixture containing cyclohexane, whose experimental distillate flow rate increases markedly with reboiler heat duty. At the same time, its bottoms product flow rate falls. The simulation results exhibit the same trend, but not the pronounced fluctuations observed in the experiment. This could be due to the small differences between the experimental and simulation solubility curves.

As regards the bottoms product (the ethanol-hydrocarbon blend destined for use as fuel), the proportions of ethanol and hydrocarbon in the simulations and experiments are similar. The water content of the bottoms also exhibits the same trend in simulation and experiment.

5. Comparison with the conventional process

Now that it has been established that the experimental and simulated results agree reasonably well and that simulations are capable of reproducing the desired bottoms product, we proceed to compare the conventional process of ethanol manufacture with the one described in this study. The conventional process to obtain pure ethanol, with benzene as entrainer, is discussed extensively in the literature [25].

For the purpose of the comparison, the simulation has been based on a flow diagram similar to the one shown in Figure 7. UNIFAC was used to model the thermodynamics of all process units.

Table 2 lists the temperatures, flow rates and compositions of the feed and product streams of the two processes that were studied with isooctane as separating agent. Streams 1 and 2 represent the water-ethanol azeotrope and hydrocarbon feed streams, respectively; they are the same in both processes. Stream 3 represents the desired product, which in the conventional process is pure ethanol, and in the proposed process is the ethanol-hydrocarbon blend. The water that has entered the system leaves it in stream WS, and corresponds to the bottoms product from the second distillation column whose function is also to remove water from the system.

If we compare the heat requirements of the process units in the two simulated processes (see Table 3) and the energy to produce anhydrous ethanol in each case (see Table 4), we obtain a threefold reduction in costs of the new process with respect to the conventional one. This leads us to believe that the new process would be a viable alternative to the conventional process.

6. Conclusions

We have shown experimentally and by simulation that it is viable both technically and economically to manufacture a ready-to-use ethanol-hydrocarbon fuel blend by azeotropic distillation.

Starting from an azeotropic mixture of water and ethanol, and with several different hydrocarbons acting as entrainers (hexane, cyclohexane, isooctane and toluene), we have demonstrated the technical viability of this process in a distillation column at the semi pilot-scale. This is based on the fact that the ethanol-hydrocarbon fuel blend obtained as final product contains less water than is stipulated by the EU legislation currently in force.

The various experiments carried out in this study show that, at a given feed rate, as the reboiler heat duty rises, the amount of water in the ethanol-hydrocarbon blend falls until the heat duty reaches a maximum value at which the distillation column ceases to operate correctly.

Conventional thermodynamic models exhibit departures from observed behavior when used to correlate experimental data, and particularly in the case of liquid-liquid equilibria in the vicinity of the plait point, which also happens to be in the vicinity of the operating range of the column. To avoid such departures, other methods of interpolating equilibrium data are required, as otherwise we would not find maximum reboiler heat duty values that result in distillation column malfunction. A simulation of the process in Chemcad with the above considerations taken into account produces results that agree well with experiment.

The simulation results of the proposed process indicate that there is a threefold reduction in heat duties compared to heteroazeotropic distillation, which produces pure ethanol.

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Tables

Table 1: Temperatures of the streams and process units for the various studied mixtures.

	Temperature (K)			
Hydrocarbon	Water-ethanol feed	Hydrocarbon feed	Decanter	Reflux
Hexane	323	331	305	329
Cyclohexane	335	339	309	338
Isooctane	347	367	318	367
Toluene	344	349	298	348

Table 2: Temperatures, flow rates and compositions of the feed and product streams for the studied processes.

Conventional Process				
Stream	**1**	**2**	**3**	**WS**
Temperature (K)	352.8	367.2	351.1	372.7
Flow rate (g/min)	4.38	$6 \cdot 10^{-5}$	4.13	0.25
Water	0.060	0.000	0.003	0.999
Ethanol	0.940	0.000	0.997	0.001
Isooctane	0.000	1.000	0.000	0.000
Proposed process				
Stream	**1**	**2**	**3**	**WS**
Temperature (K)	352.8	367.2	346.4	372.7
Flow rate (g/min)	4.38	39.73	43.81	0.26
Water	0.060	0.000	0.000	0.999
Ethanol	0.940	0.000	0.094	0.001
Isooctane	0.000	1.000	0.906	0.000

Table 3: Comparison of the energy needs of the simulated process units

Process unit	Column 1 (W)	Condenser (W)	Column 2(W)
Conv Process	275.15	-986.04	711.48
New Process	81.42	-370.64	256.85

Table 4: Comparison of heat duties in the production of anhydrous ethanol

Process unit	Column 1 (W/gEtOH)	Condenser (W/gEtOH)	Column 2 (W/gEtOH)
Conv Process	66.82	-239.47	172.79
New Process	19.83	-90.29	62.57

Figure Captions

Figure 1: Schematic representation of the equipment used

Figure 2 a): Mixture containing hexane. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

Figure 2 b): Mixture containing hexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and hexane in the column bottoms

Figure 2 c): Mixture containing hexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.

Figure 3 a): Mixture containing cyclohexane. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

Figure 3 b): Mixture containing cyclohexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and cyclohexane in the column bottoms.

Figure 3 c): Mixture containing cyclohexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product

Figure 4 a): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

Figure 4 b): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and isooctane in the column bottoms

Figure 4 c): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.

Figure 5 a): Mixture containing toluene. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

Figure 5 b): Mixture containing toluene. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and toluene in the column bottoms

Figure 5 c): Mixture containing toluene. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.

Figure 6: Schematic diagram for the Chemcad simulation.

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404 **Figure 7**: Schematic diagram used to compare both processes.
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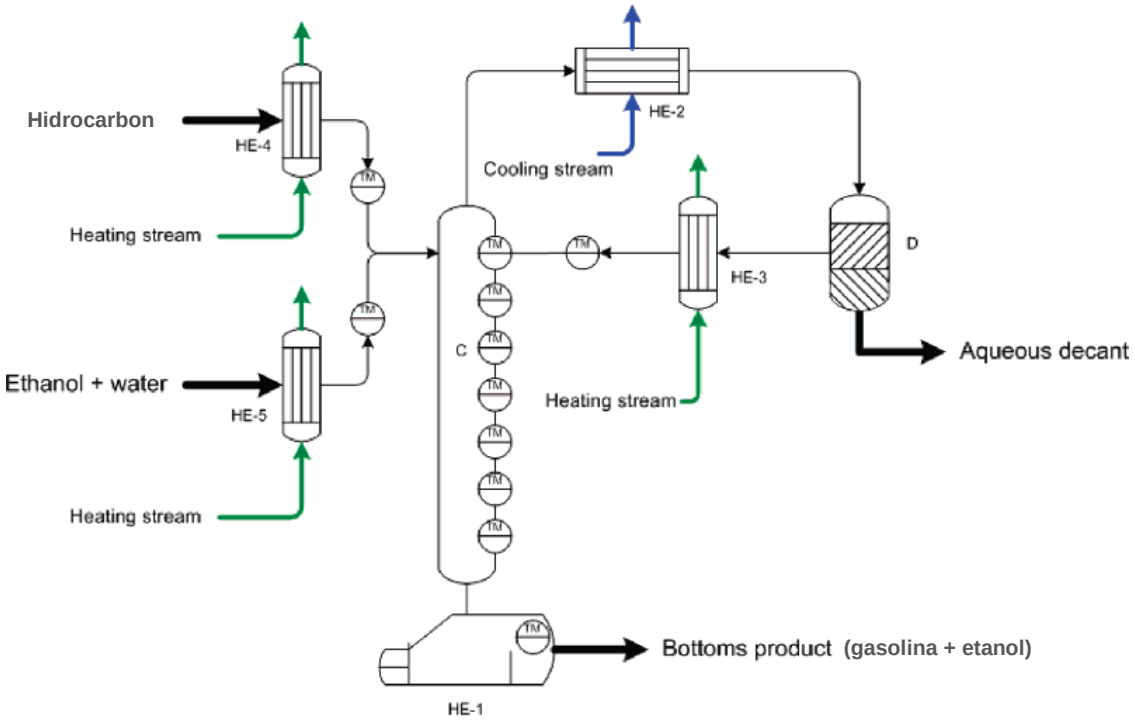


Figure 1: Schematic representation of the equipment used

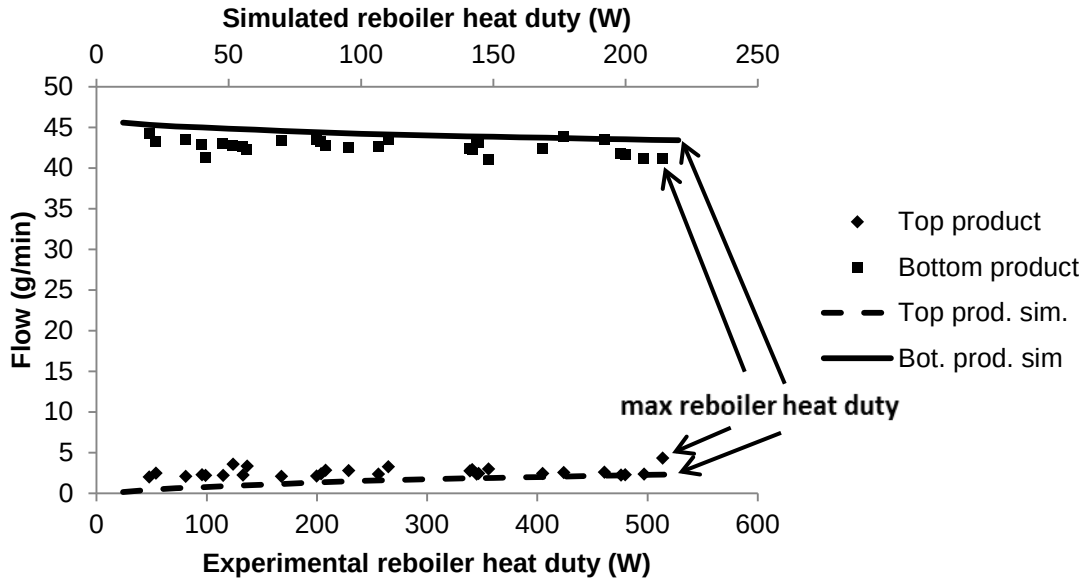
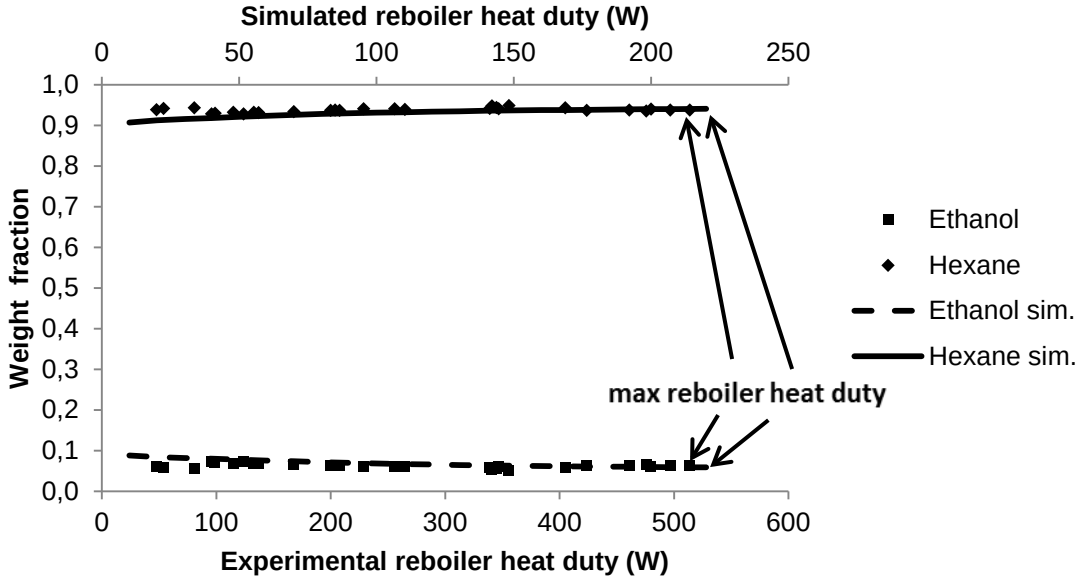


Figure 2 a): Mixture containing hexane. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates



414
415 **Figure 2 b):** Mixture containing hexane. Plot of several variables against reboiler heat duty:
416 Experimental and simulated composition (% w/w) of ethanol and hexane in the column
417 bottoms
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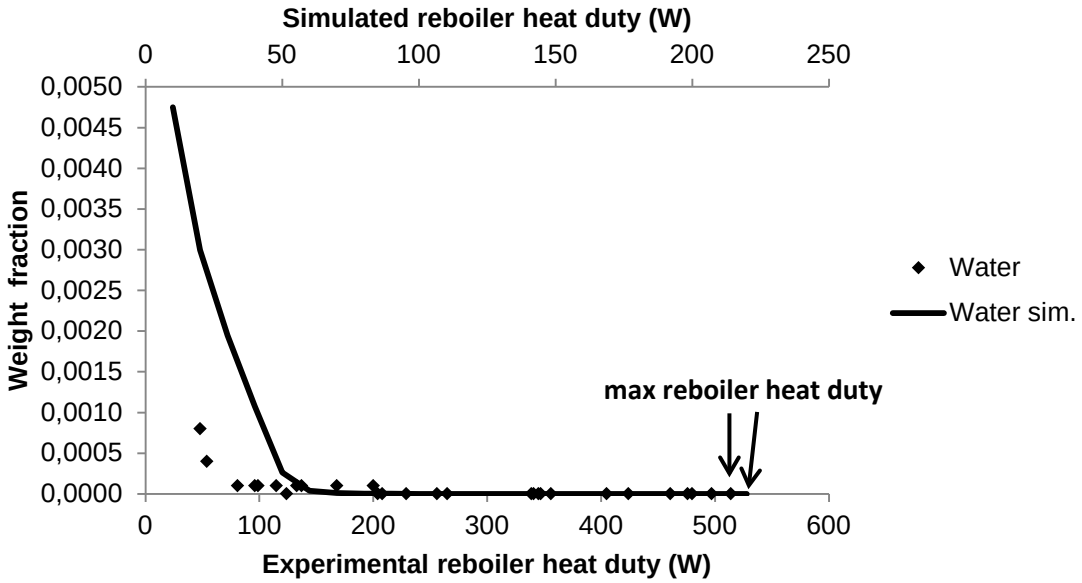
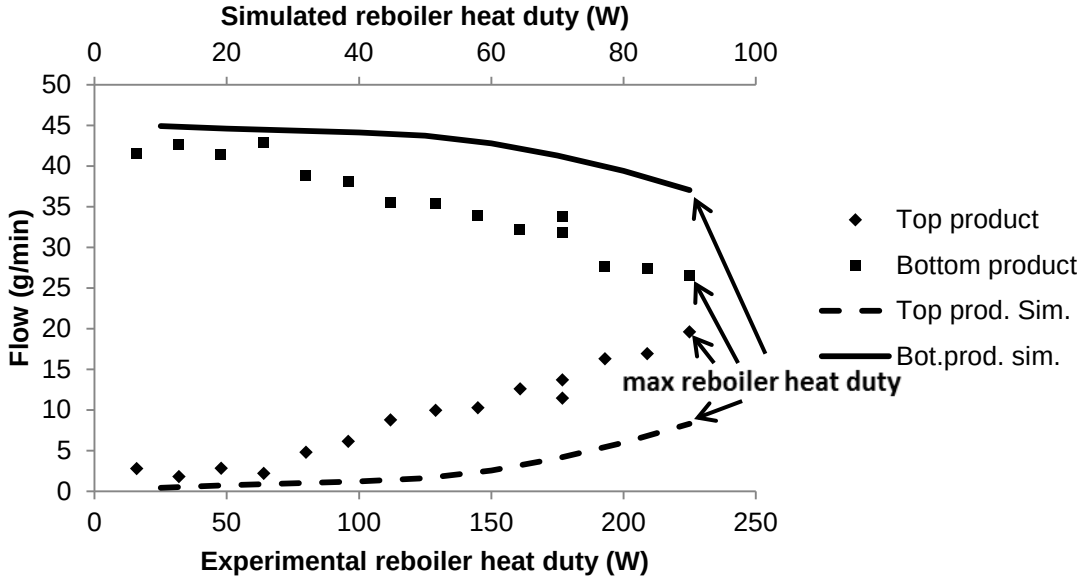


Figure 2 c): Mixture containing hexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.



423
424 **Figure 3 a):** Mixture containing cyclohexane. Plot of several variables against reboiler heat
425 duty: Experimental and simulated top and bottoms product flow rates
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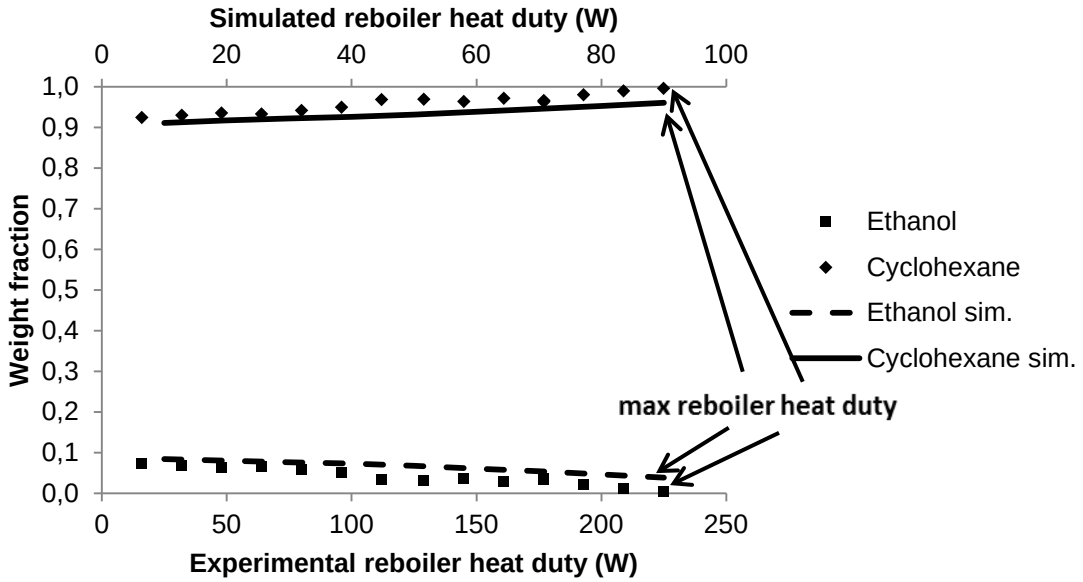


Figure 3 b): Mixture containing cyclohexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and cyclohexane in the column bottoms.

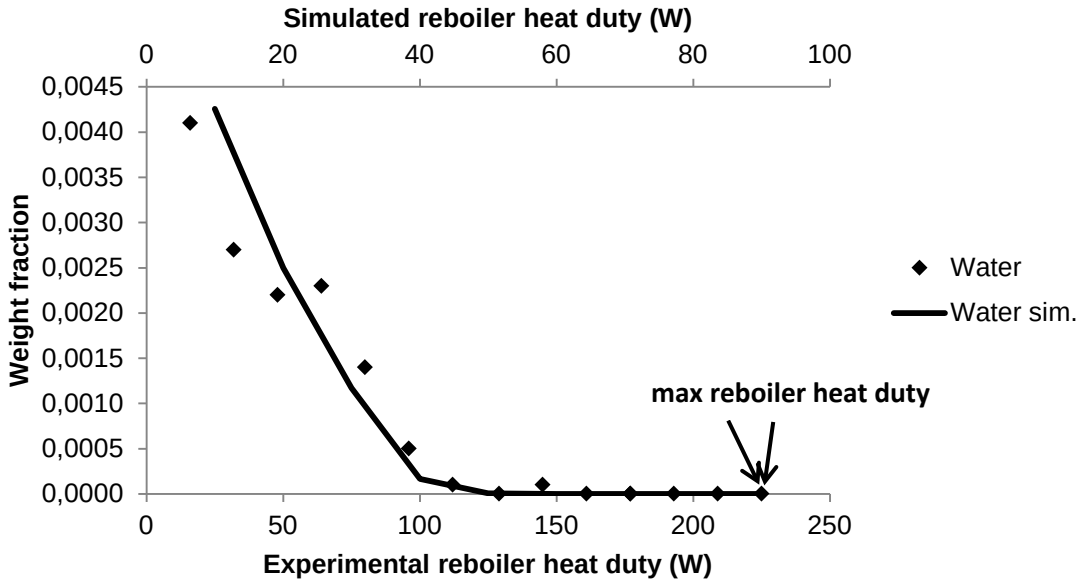


Figure 3 c): Mixture containing cyclohexane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.

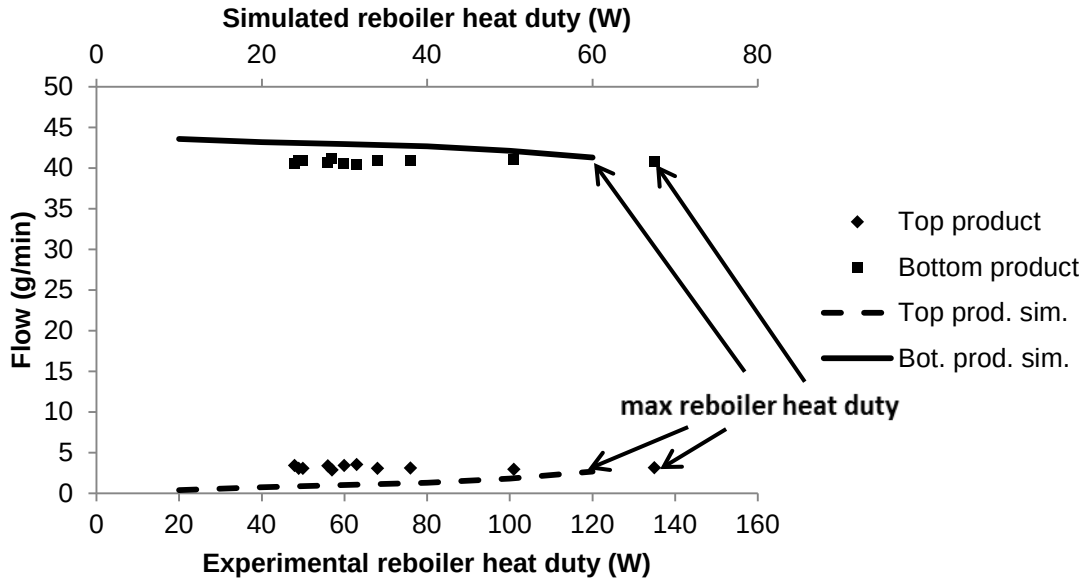


Figure 4 a): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

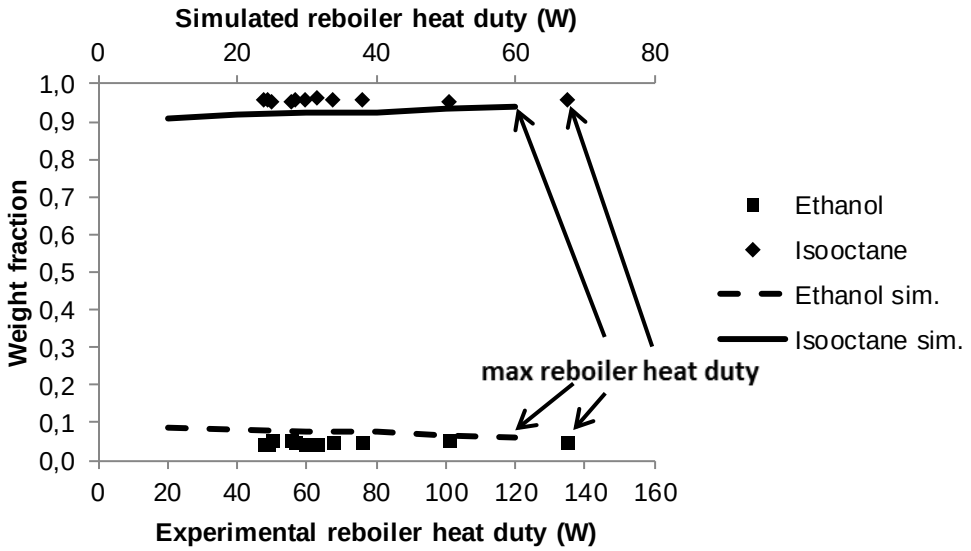


Figure 4 b): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and isooctane in the column bottoms

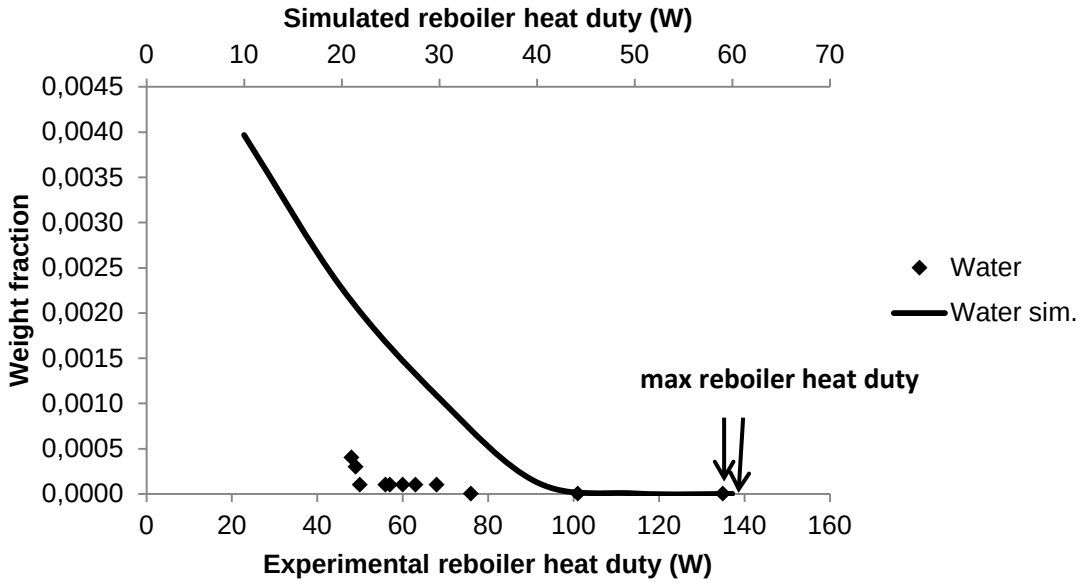


Figure 4 c): Mixture containing isooctane. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of water in the bottoms product.

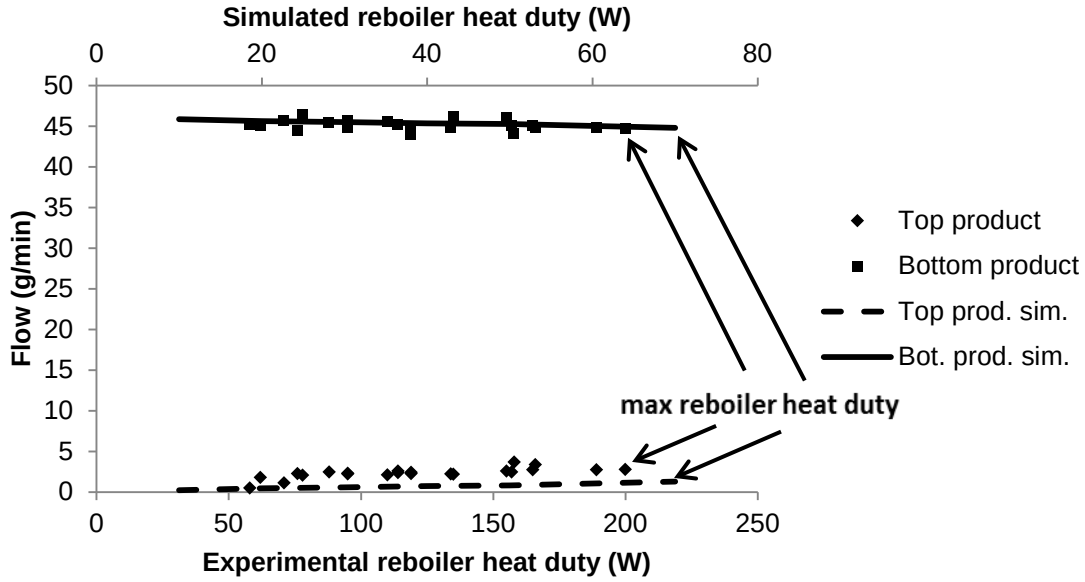


Figure 5 a): Mixture containing toluene. Plot of several variables against reboiler heat duty: Experimental and simulated top and bottoms product flow rates

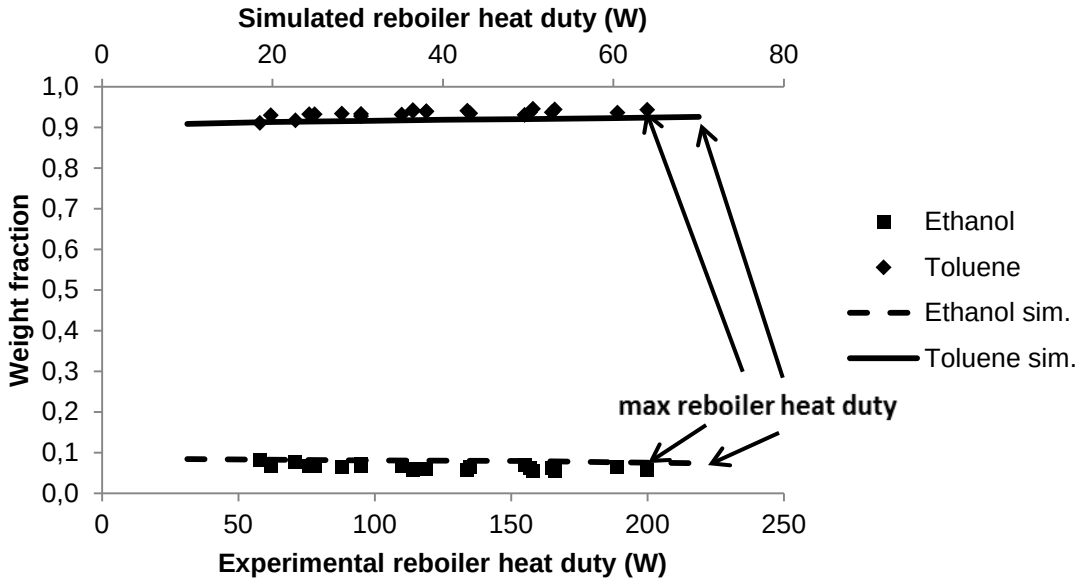
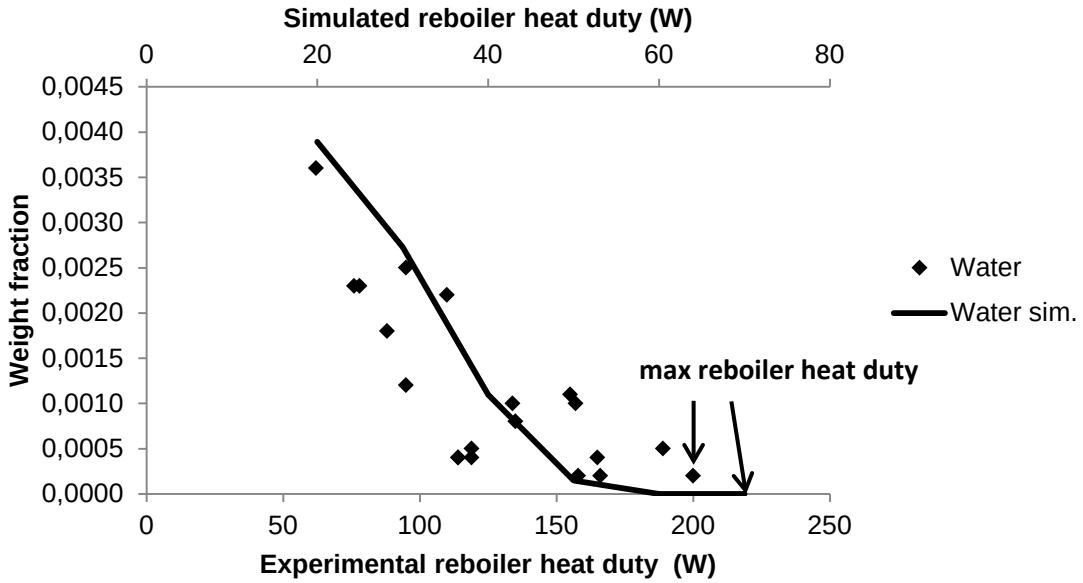


Figure 5 b): Mixture containing toluene. Plot of several variables against reboiler heat duty: Experimental and simulated composition (% w/w) of ethanol and toluene in the column bottoms



460
461 **Figure 5 c):** Mixture containing toluene. Plot of several variables against reboiler heat duty:
462 Experimental and simulated composition (% w/w) of water in the bottoms product.
463
464

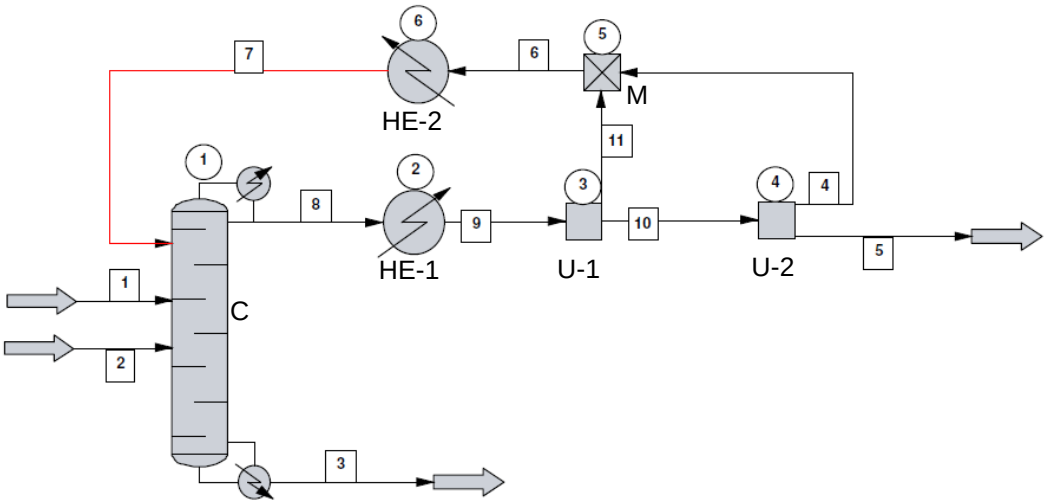


Figure 6: Schematic diagram for the Chemcad simulation.

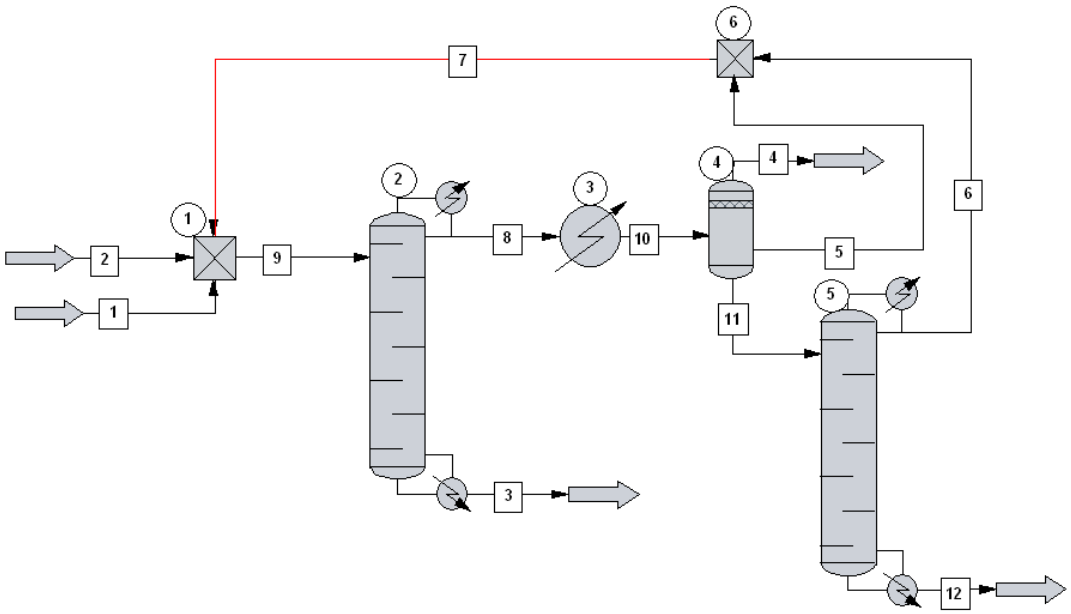


Figure 7: Schematic diagram used to compare both processes.