

## Optimisation of a Bio-ethanol Purification Process Using Conceptual Design and Simulation Tools

Patricia M. Hoch,<sup>a</sup> José Espinosa,<sup>b</sup>

<sup>a</sup>*PLAPIQUI-UNS-CONICET, Camino La Carrindanga km 7-8000, Bahía Blanca,  
Argentina*

<sup>b</sup>*INGAR-CONICET-UNL, Avellaneda 3657, S3002 GJC Santa Fe, Argentina*

### Abstract

In this work, we propose an evolutionary optimisation procedure which intensively uses both conceptual and rigorous models for the design and simulation of unit operations involved in a bio-ethanol purification process. While conceptual models are used to determine initial values for design and operating variables of a given operation unit, rigorous simulation departing from initial estimates generated at conceptual design level enables to remove simplifying assumptions, interconnect equipments and calculate operating and investment costs.

Once an initial design of the purification plant is obtained, opportunities of improvement are easily recognized and then tested by performing the design and simulation steps until a cost-effective bio-ethanol purification plant is achieved.

**Keywords:** Bio-ethanol Purification Plant, Evolutionary Optimisation, Conceptual Design, Rigorous Simulation.

### 1. Introduction

Optimisation of both design and operation variables of a bio-ethanol purification plant intended to produce fuel grade ethanol is a challenge to make the bio-fuel a realistic alternative in the energy market.

Given the plant complexity and the high non-linearity of the corresponding models, we propose an evolutionary optimisation procedure which uses intensively both conceptual and rigorous models for the design and simulation of unit operations. It is quite difficult to obtain an initial estimation of the total number of trays of the main distillation column, placement of feed and side streams, and steam flow rate in the simulation environment, but this task is easily accomplished in the conceptual design environment.

In addition, convergence of the rigorous model is enhanced by using initial estimates of internal profiles generated at the conceptual design level despite of the highly non-ideal behaviour of the multicomponent azeotropic mixture. Applying the same philosophy to estimate initial values for design and operating variables of the whole process, optimal values for a cost-effective bio-ethanol purification plant are reported.

The methodology is applied to the purification process for a feed leaving the fermentation step of a conventional corn dry-grind processing facility producing 24 million liters/year of ethanol and 19 million kg/year distiller's dry grains with solubles (DDGS). The feed to the purification plant (22170 kg/h) is mainly composed by ethanol (10.80 % w/w) and water (88.98 % w/w) with traces of methanol (0.0226 %) and fusel (0.2009 %). A simplified flow diagram is shown in Figure 1.

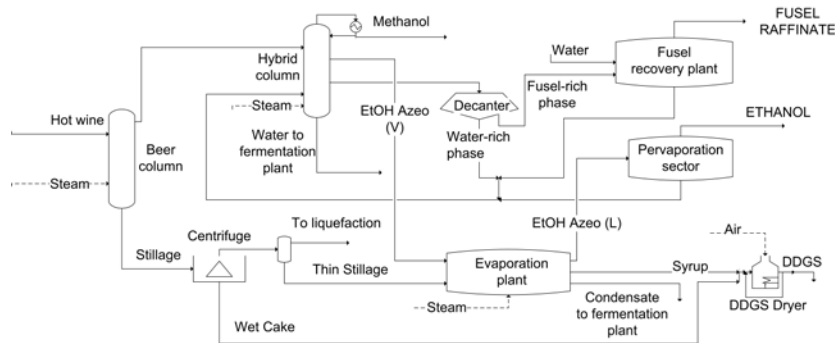


Figure 1. Simplified flow diagram of a bioethanol purification plant.

## 2. Initial Design

### 2.1. Beer Column

The vapour stream leaving the stripping column captures nearly all of the ethanol and components in traces produced during the fermentation step. The minimum energy demand of the process (i.e., minimum reboil ratio) is calculated through the lever arm rule by setting the bottom product as high purity water and the composition of the vapour stream as the corresponding to the vapour in equilibrium with hot wine. In other words, a pinch at the top of the stripping column is considered.

In order to obtain a feasible design the following steps are performed: i) determine the maximum feasible separation and minimum energy demand ( $s_{\min}$ ) by applying pinch theory. For this task, it is required an equilibrium calculation that can be performed in a conceptual model framework like DISTIL (Hyprotech, 1999); ii) calculate the reboil ratio  $s = 1.05 s_{\min}$ ; iii) set a value for the number of stages  $N$ ; iv) simulate a stripping column with reboiler first and then replace the reboiler with steam taking into account the reboiler duty and the latent heat of condensation of steam. This task can be done in a simulation framework like Hysys (Hyprotech, 1999).

Few simulations were needed to achieve a quasi-optimal column design with 38 equilibrium stages, a column diameter of 0.9 m, a section pressure drop of 34.7 kPa and a steam flow rate of 3600 kg/h. The values obtained for the number of equilibrium stages and steam demand, together with the composition of ethanol in the outlet stream (51.52 % w/w) agree well with results presented in Kwiatkowski et al. (2006).

### 2.2. Hybrid Column

As a multicomponent system formed by ethanol and water with traces of methanol and fusel is to be separated into a single column, a three steps conceptual design and rigorous simulation process is proposed. First, ethanol and methanol are lumped into one pseudo-component in order to obtain a first estimation of steam flow rate, number of stages necessary to separate an ethanol-rich stream from a fusel-rich stream, feed stage and side stream location (DISTIL). After rigorous simulation of the quaternary mixture in Hysys, separation between a methanol-rich stream and an ethanol-rich stream is considered by taking into account the distillation line departing from the composition of the distillate (DISTIL). Finally, both columns are integrated into a single one (Hysys).

#### 2.2.1. Side-Stream Column

In order to obtain a feasible design the following steps are performed: i) calculate  $s_{\min}^{(1)}$  for the separation ethanol/water/1-pentanol (DISTIL), ii) estimate the number of

equilibrium stages  $N_{\text{stages}}^{(t)}$ , feed stage  $N_{\text{feed}}^{(t)}$  and side stream location  $N_{\text{Side Stream}}^{(t)}$  for  $s^{(t)} > s_{\text{min}}^{(t)}$  (DISTIL), iii) simulate the quaternary system (Hysys) for the design and operating variables obtained in step ii), iv) calculate the steam flow rate  $V_{\text{steam}}^{(t)}$  through the energy balance and simulate the column without reboiler (Hysys), v) simulate the system side-stream column plus decanter and water-rich phase recycle (Hysys).

Table 1 presents the results of the conceptual design performed in DISTIL (steps i) and ii)) for the ternary mixture ethanol/water/1-pentanol, with the last component used to approximate the behaviour of a fusel component. Compositions of both ethanol and fusel in the bottom stream were selected taking into account the behaviour of the residue curve (DISTIL) corresponding to the liquid in equilibrium with the vapour feed in the neighbourhood of water vertex. As both for the ternary and quaternary mixture, the residue curve approaches the water vertex with compositions of ethanol above the mole fraction of fusel, the selected bottom composition reflects this behaviour. Figure 2(a) shows the corresponding composition profile in the composition simplex. The figure also shows a distillation boundary departing from the azeotrope ethanol-water and ending at the hetero-azeotrope water-fusel. The side stream is located inside the liquid-liquid gap as this stream will be separated in a decanter into a fusel-rich phase (feed to the fusel plant) and a water-rich phase (recycle to column).

Table 1. Results for the ternary system ethanol/water/1-pentanol (DISTIL).

	Product Compositions, r, s and N
Feed (vapor)	[0.29798, 0.699132, 0.002888]
Distillate	[0.74144, 0.25850, 6.0 E-05]
Side Stream	[0.05115, 0.91442, 0.03443]
Bottom	[9.6 E-06, 0.99999, 4.0 E-07]
$R_{\text{min}}^{(t)} / S_{\text{min}}^{(t)}$	2.839 / 1.00
$R^{(t)} / S^{(t)}$	2.839 / 1.00
$N_{\text{stages}}^{(t)}$ [including condenser and reboiler]	17 [0 + 1-15 + 16]
$N_{\text{feed}}^{(t)} / N_{\text{Side Stream}}^{(t)}$	4/11

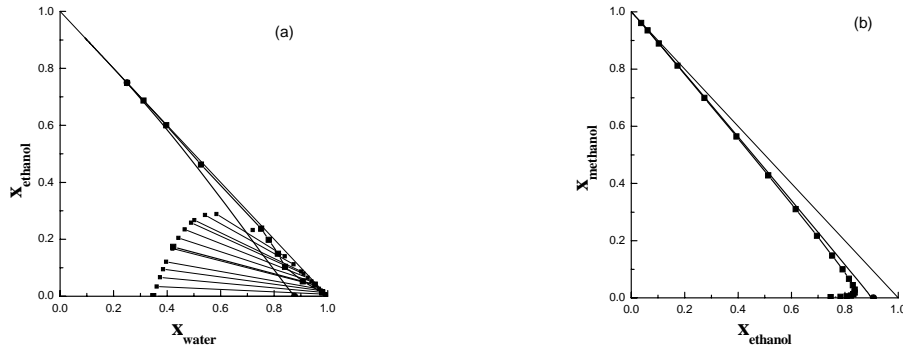


Figure 2. (a) Internal profile in the composition simplex corresponding to the base design with phase separator and water-rich phase recycle (Hysys); (b) Distillation lines corresponding to the distillate composition of the methanol column. System methanol/ethanol/water+1-pentanol at 101.3 kPa (DISTIL).

It is noteworthy that minimum reboil ratio for the given separation does not require an infinite number of stages. This behaviour can be explained in terms of bifurcation of adiabatic profiles and it will be subject of further analysis in a next contribution.

### 2.2.2. Methanol Column and Hybrid Column

The distillate stream of the side-stream column contains small amounts of methanol that must be separated from ethanol to agree with stringent quality standards (0.1 % V/V methanol in dehydrated ethanol at 20 °C). At a first glance, this separation could be performed in another distillation column. A different alternative would be to integrate this column with the side-stream column. The distillate line (DISTIL) corresponding to the distillate composition of the side-stream column shown in Figure 2(b) resembles the behaviour of the internal profile at total reflux and it can be considered as a good approximation to the actual operation of the methanol rectifying column, as the distillate flow rate of this column is low enough to produce a high reflux ratio. Therefore, it is possible to estimate the optimal number of stages in the rectifying section of the hybrid column as the number of trays necessary to separate the methanol in excess from the ethanol-rich stream. As shown in Figure 2(b), the methanol-rich distillate stream will also contain small amounts of ethanol and water due to the distillation line running from pure methanol to ethanol-water azeotrope.

Figure 3 shows the internal profile in the composition tetrahedron after simulation of the hybrid column in Hysys. A loss of about 0.18 % w/w of ethanol in methanol-rich distillate occurs. The side streams are located near the maximum in ethanol (ethanol-rich stream, 88.98 % w/w) and fusel (fusel-rich stream, 18.5 % w/w), respectively. The column has 35 equilibrium stages, a column diameter of 1.372 m, a section pressure drop of 11.8 kPa and a steam flow rate of 1800 kg/h. The vapour ethanol-rich stream is diverted to the first effect of the evaporation sector to provide heating while minimizing the steam demand of the plant. The condensed ethanol-rich stream is then fed to the pervaporation sector to remove the excess water.

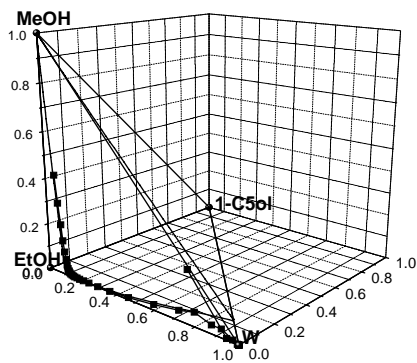


Figure 3. Internal profile in the composition tetrahedron corresponding to the hybrid column with phase separator, fusel plant and pervaporation sector.

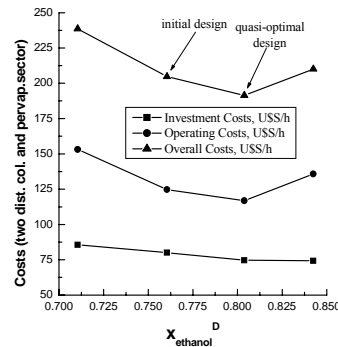


Figure 4. Overall investment and operating costs for the two distillation columns and pervaporation sector versus ethanol mole fraction in the distillate of the hybrid column.

### 2.2.3. Fusel Plant and Pervaporation Sector

The fusel-rich stream leaving the decanter is fed to the fusel sector where the stream is washed with water to recover about 96 % of the incoming ethanol. The resulting water-rich stream is recycled to the hybrid column. To do this, an overall amount of 363 kg/h wash-water and seven separation steps are necessary. The conceptual design of a cross-flow operation is performed using DISTIL, while process simulation is done in Hysys. A conceptual design of pervaporation (PVA/PAN MOL 1140 membrane by GFT, Germany) following the model proposed by Vier (1995) and Bausa and Marquardt (2000) was implemented in Delphi environment (Borland, 1997) to determine pseudo

optimal operating values for both maximum temperature (90 °C) and permeate pressure (2.026 kPa). The model was also implemented in Hysys as a user operation extension, which calculates the required membrane area  $a_{req}$  as  $a_{req} = 1.25 a_{min}$  (Bausa and Marquardt, 2000). Both heat and refrigeration duties are calculated in Hysys from the energy balance. Behaviour of trace components is taken into account by using water-alcohol separation factors. Approximate values were taken from D. Van Baelen et al. (2005). High purity bioethanol (99.5 % v/v) is obtained with an actual membrane area of 930 m<sup>2</sup>, while the water-rich permeate is recycled to the hybrid column.

**2.2.4. DDGS Plant**

The coproduct plant is formed by a decanter centrifuge which separates the bottom stillage from the beer column in a wet cake (35 % solids, 2683 kg/h water) and a thin stillage (18443 kg water/h). Approximately 4626 kg water/h is recycled into the second step of the liquefaction process, while 13817 kg water/h is feed to a three-effect evaporator. The resulting syrup (35 % solids, 1287 kg water/h) is mixed with the wet cake coming from the centrifuge and sent to a rotary drum dryer. While the multiple effect evaporator is simulated in Hysys, only mass and energy balances for the dryer are incorporated in Hysys. The conceptual design of the dryer is performed according to the method presented in Ulrich and Vasudevan (2004) and data from National Corn-To Ethanol Research Center (2005-2007). Table 2 summarizes the investment and operation costs of the DDGS sector. The operation cost of 35.9 \$/ton DDGS agrees well with the value reported by Batelle Memorial Institute (2005).

Table 2. Operation (\$/ton DDGS) and Investment (\$) Cost corresponding to DDGS plant. [\*] kg natural gas/ kg of water evaporated. [\*\*] Overall operating costs = 34.55 \$/ton DDGS

Item	Characteristics	Invest./Op. Cost Ulrich & Vasudevan	Invest./Op. Cost [**]; Batelle M. I. [***]
Rotary Dryer	D [m]= 1.664 L [m]= 13.31 $\tau$ [min]= 19.6 rpm= 2.869 $Q_{air}$ [kg/h]= 39150 $\eta_{Nat. Gas}$ = 0.048 [*]	1.39 E06/19.74	1.57 E06
Evaporator	Area <sub>overall</sub> [m <sup>2</sup> ]= 940 Pressure[kPa]= from 10-30 kPa $Q_{steam}$ [kg/h]= 2700	1.523 E06/16.16	1.47 E06
Decanter Centrifuge		1.07E06[***]/not calculated	1.07 E06

**3. Evolutionary Optimisation**

Once an initial design is obtained, the following improvement opportunities were tested: i) design of the beer column in the neighbourhood of minimum energy demand (done from the very beginning of design process), ii) heat integration between the hybrid column and evaporation first effect (1270 kg/h of steam are saved), iii) heat recovery from the hot air leaving the dryer (saving 0.014 kg natural gas/kg water evaporated). Finally, a change in distillate composition of the hybrid column is proposed in order to capture the trade-offs between distillation and pervaporation costs. Resorting again to the conceptual design of the hybrid column for a set of distillate compositions, results

shown in Figure 4 are obtained. The decrease of investment costs for distillate compositions richer in ethanol is related to a decrease in the membrane area needed to obtain dehydrated bioethanol ( $a_{\text{req}}=848 \text{ m}^2$ ). Table 3 shows both investment and operating costs of the quasi-optimal plant. All cases include improvements i), ii) and iii) mentioned above. Investment and operation costs are reduced by 6.64 % with respect to the base case and by 11.48 % with respect to the worst case analyzed. Cost coefficients used to obtain the reported values are  $C_{\text{steam}}=2.396\text{E-}2$ ,  $C_{\text{water}}=5.73\text{E-}3$ ,  $C_{\text{refr}}=3.824\text{E-}2$ ,  $C_{\text{electr}}=0.08$  all in [\$/kWh],  $C_{\text{nat gas}}=289 \text{ \$/tn}$ ,  $C_{\text{memb repl}} = 400 \text{ \$/m}^2$ .

Table 3. Overall costs for a bioethanol plant producing 24 million liters/year. The facility considers both the ethanol and co-product processing plants.

	Investment, \$	Operating \$/h	Investment, \$/h	Total, \$/h
DDGS Plant	3.985 E+06	96.37	81.07	177.44
Separation	3.768 E+06	117.96	76.66	194.62
Total	7.753 E+06	214.33	157.73	372.06

#### 4. Conclusions

A cost effective design for a bio-ethanol separation plant using conceptual design followed by rigorous simulation is found. The minimum in the operation costs corresponds to a minimum in the steam flow rate of the hybrid column (1600 kg/h). The minimum in steam flow rate can be only explained by the presence of the fusel component, which influences both the energy demand and feasible products of the process. Therefore, designs based on the binary system ethanol-water do not represent the system behaviour in an accurate way.

#### 5. Acknowledgements

This work was supported by UNL, UNS, CONICET and ANPCyT from Argentina.

#### References

- Batelle Memorial Institute, Pacific Northwest Division, 2005, Quantifying Biomass Resources for Hydrothermal Processing II.
- Bausa, J. and W. Marquardt, 2000, Shortcut Design Methods for Hybrid Membrane/Distillation Processes for the Separation of Nonideal Multicomponent Mixtures, *Ind. Eng. Chem. Res.*, 39, 1658-1672.
- Borland International Inc., 1997, Scotts Valley, USA, Delphi 3 User Manual.
- Hyprotech Ltd., 1999, Calgary, Canada, Hysys & Distil User Manuals.
- Kwiatowski, J. R.; McAloon, A. J.; Taylor, F. and D. B. Johnston, 2006, Modeling the Process and Costs of Fuel Ethanol Production by the Corn Dry-Grind Process, *Industrial Crops and Products*, 23, 288-296.
- National Corn-To Ethanol Research Center, 2005-2007, Utilizing the National Corn-to-Ethanol Pilot Plant to Develop a Predictive Model for Distillers Dried Grain for the Fuel Ethanol and Animal Feed Industries.
- Ulrich, G. D. and P. T. Vasudevan, 2004, *Chemical Engineering Process Design and Economics, A practical guide*. 2nd ed., Process Publishing, Durham, New Hampshire.
- Van Baelen, D.; Van der Bruggen, B.; Van den Dungen, K.; Degreve, J. and C. Vandecasteele, 2005, Pervaporation of water-alcohol mixtures and acetic acid-water mixtures. *Chemical Engineering Science*, 60, 1583-1590.
- Vier, J., 1995, Pervaporation azeotroper wässriger und rein organischer Stoffgemische-Verfahrensentwicklung und -integration, Ph. D. Thesis, Institut für Verfahrenstechnik, RWTH Aachen, Shaker Verlag, Aachen, Germany.