# Designing Experiments with Aspen HYSYS Simulation to improve Distillation Systems

## Insights from a Chemical Engineering Course

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*Abstract*—**Designing experiments by simulation with process simulators, e.g. Aspen HYSYS, can be very effective in the optimization of several chemical engineering systems. It is important to enhance students' skills highlighting the value of applying problem specific knowledge, simulation tools and sound statistical techniques. This paper addresses the use of Aspen HYSYS by Portuguese chemical engineering master students to model distillation systems together with statistical experimental design techniques in order to optimize the systems. The paper presents a procedure followed in two projects beginning with the selection of two case studies from the literature, which the goal is to further improve with the help of design of experiments. The paper presents the work developed by the students in order to model steady-state and dynamic processes and optimize the distillation systems emphasizing the benefits of the simulation tools and statistical techniques in helping the students learn how to learn. Students strengthened their domain specific knowledge and became motivated to rethink and improve chemical processes in their future chemical engineering profession. The main results and conclusions provide a strong incentive from the teachers´ perspective to a wider use of this kind of procedure by chemical engineering master students.**

***Keywords— Distillation, Aspen HYSYS, Factorial design, Active learning, Problem solving***

## I. Introduction

The impact that chemical processes have on the environment and on the economy are of growing concern to the public, the chemical industries, and regulatory agencies. Chemical engineering students must develop skills with the goal of designing, operating, optimizing and maintaining chemical processes in such a way that they are valuable to society, environmental benign and economically viable [1,2]. Students need to understand the design and operation of chemical processes, how changes in operations and in the environment will affect outcomes, and the actions needed to improve the performance of a process increasing sustainability.

Separation is one of the fundamental processes in chemical engineering and most chemical processes involve separation. Therefore, due to their popularity in the chemical process industries, looking at the design and optimization of separation systems is very important [3].

Distillation is the main separation process used in chemical industries for separation of liquid mixtures due to its versatility and ability to large-scale production. This makes, and will continue to make, distillation the main source for many separation processes. Distillation columns are the most commonly used separation units in a refinery and generally in petrochemical and chemical industries [4]. However, distillation is a highly energy intensive process which can make operating costs a concern and also, the environmental impact of such highly energy intensive process a key issue.

One of the current and future challenges is to study alternatives in terms of configuration and design of distillation structures that allows improving the efficiency of the process and reducing the energy consumption in order to meet the demands of the energy conscious society [5].

However, improving the process is not straightforward because the columns may have many different configurations with diverse operating objectives. These leads to several operational degrees of freedom and distinct dynamic behaviours making it difficult to optimize and control, reason why the optimal configuration of distillation systems is a major problem in the design of chemical processes [6].

The use of chemical process simulation software is very important to optimize chemical processes in industry and, in the specific case of distillation, enables a simple and quick conceptual design of distillation structures in order to make separations of chemical mixtures. It is of utmost importance that chemical engineering students known how to use simulation software to the rapid conceptual design of distillation schemes for the separation of chemical mixtures. The students are engaged to use software like Aspen HYSYS in a problem based learning environment to assess the limits of feasible separations for individual columns and also to design and operate advanced distillation structures for the separation of mixtures with non-ideal vapour-liquid equilibrium. The students should also understand the dual perspectives of steady state simulation and also dynamic simulation. It is worth to mention that traditional distillation design textbooks have focused predominantly on the steady state design and economics aspects of distillation. However, it is also desirable that the students learn how to deal with such issues as dynamic simulation in face of disturbances in order to develop effective control structures [7].

The design of experiments (DOE) is a strategy of planning, conducting, analysing, and interpreting experiments, in order to take valid conclusions effectively and economically. However, the application of DOE by chemical engineers in industry is quite limited due to the lack of statistical knowledge in experimental design techniques [8]. In order to bridge this gap it is crucial that chemical engineering students use DOE and realize the benefits.

In this paper we explore the use of Aspen HYSYS to model the distillation systems and the methodology of experimental design in order to improve the systems. In two different projects, a case study reported in the literature was presented to the students. The idea is to use a case study reported in the literature as a starting point. This can be a very challenging learner based teaching method bringing new insights into the problems allowing students to explore new issues in an active way providing the opportunity for the development of key skills such as problem solving in real world situations. In the next section we present the advantages of a learner centred approach supported by computer aided learning packages and statistical tools and the relative merits of the active learning approach concerning the engagement of the students with the project and acquisition of skills and competences by means of an active learning process. The section 3 explains the motivation for integrating DOE with simulation by Aspen HYSYS to improve distillation systems. The methodology used in the projects developed in our chemical engineering master course is also explained in section 3 and two case studies each one developed by one master student in his/her final project is presented in section 4 along with the main results reached by the students. Section 5 presents the evaluation of the projects and the impact on students learning. The paper concludes with the main advantages of the methodology from the students' point of view as well as some insights from the teachers' perspective.

## II. Student Centred Approach Supported by Computer Simulation Packages and Statistical Tools

The teaching in a chemical engineering course needs to reflect the challenges that the chemical engineers will face in industry. The final project of a second cycle degree can be an opportunity to instigate the students' way of thinking from the role of being a student to an engineer´s thinking applying known scientific concepts for practical solutions. After graduate second cycle degree a chemical engineer should be able to learn on his/her own and have an understanding of the impact of engineering solutions in an economic, environmental, and societal context. One effective way to have students learn how to learn is to have students involved in projects which usually require significant faculty time reason why the final project is by no doubt very useful to simulate what engineers do in practice.

Recent proliferation in computer simulation and modelling makes it simple to use computer aided learning into teaching practices at chemical engineering. According to Glassey et al. [9] computer aided learning packages can be useful tools in supporting the development of important professional attributes as they enable students to explore and gain experience of new software environments in subject-specific context. The many advantages of chemical process simulation software makes it particularly appropriate to support student centred learning. Chemical process simulators can facilitate experimentation in real world settings. The Aspen HYSYS software aids students in learning how to use a chemical process simulator and how a process simulator is able to model quite a lot of chemical process units. The use of statistical tools along with chemical simulation packages allows to closely relating statistics as a scientific tool for solving real world problems. A review of the literature in statistical education shows that students may learn more readily with a student centred approach as compared to the traditional passive lecturing style [10-12]. Some authors realized that students sometimes develop misconceptions while studying statistics and so it is important that students might be confronted with their erroneous thoughts to remedy such misconceptions [13,14].

Placing the students in the centre of the learning process is a very enriching learning model [15]. Lambert and McCombs in 1998 [16] and Bransford et al. in 1999 [17] have published books with relevant information about the superiority of student centred and active learning approaches when compared to a conventional teacher centred approach. In a student-centred approach the teacher provide the students with an opportunity to learn independently and coaches the students with the skills they need to do so effectively. Handelsman et al. [18] stated that engaging students in discovery and scientific process improves learning and knowledge retention. Some studies allow concluding that in an active learning environment students' get much greater conceptual understanding, more independence and greater confidence [19].

In the master project students need to do a variety of tasks by themselves and must be engaged with the project in order to be successful. The teacher that is coaching the student must place the responsibility for learning in the shoulders of the student and so the focus should be on what the student do and what the teacher want him/her to accomplish. Chemical process simulation packages and statistical techniques are useful tools to engage students with the problem or scenario to study increasing students´ technical knowledge as well as competences in domain specific problem solving. By using this type of tools students learn to be conscious of what information they already know about the problem in hands, what information they need to know to solve the problem and the strategies to use. The articulation of such thoughts helps students become more effective problem solvers and self-directed learners [20].

## III. Integrating DOE with Simulation by Aspen HYSYS to Improve Distillation Systems

Throughout the chemical engineering master course in our institution the students do not usually interact with design of experiments (DOE) in laboratory sessions or in pilot tests mainly due to budgetary constraints as well as the time needed to perform the experiments, despite the importance of such tools. We think that the integration of experimental design tools with process simulators (e.g. Aspen HYSYS) can overcome this gap because students learn how to use this kind of statistical tools in a simple manner performing statistical designed experiments by simulation, which is an economic and fast way to make the experiment. A very effective way to illustrate the advantages of statistical designed experiments combined with process simulators is to introduce the approach to the students and let them use and take their own conclusions. The students use as a starting point case studies of distillation systems described in the literature and are able to understand, with real case studies, the benefits of DOE concerning the optimization of such distillation systems.

The most common experimental methodology that engineers use is the one factor at time (OFAT) procedure holding all factors constant except one. In this procedure we vary this factor to find its best level and then we hold this factor constant and choose another factor to vary. We repeat the procedure until all factors have been varied. Many engineers that perform OFAT experimentation fail to realize the advantages of statistically designing experiments until they use the approach and see the benefits of manipulate all the factors at a time and identify important interactions that may be missed when experimenting with one factor at a time [21,22]. There are some examples of student engagement with statistical design of experiments by active learning projects [23].

Designed experiments are indeed a powerful approach to improving a system. To use the approach students must have, in advance, a clear idea of the objective of the experiment and the guidelines to use for designing the experiments [24,25].

Through the use of real cases, the students of our chemical engineering master course involved in those projects are encouraged by hands on approach procedure to effectively use DOE for planning and conducting the experiments, and also analysing the results statistically. The main objective is that the students are able to select the factorial designs to use in order to study alternatives in terms of configuration and design of distillation structures that allow improving the efficiency of a given process and see the benefits of the methodology using real industrial examples. Throughout the entire process, it is important to keep in mind that experimentation by simulation is an important part of the learning process. The students state the problem and acquire knowledge about the important factors, the ranges over which these factors are to be varied, the appropriate number of levels to use and the proper units of measurement of such variables. Initially students do not know perfectly the answers to those questions but they learn about them as they are going along. The students actively learn as the experimental process progresses, and often need to drop some factors, add others, change the region of exploration for some factors and so on, experimenting sequentially in order to reach an optimal solution. After reaching the optimal conditions in a steady state simulation the students are encouraged to implement a dynamic simulation of the solution obtained to better validate the solution. The dynamic simulation implies the determination of the real dimensions of all equipment, the choice of the control variables and the implementation of the control loops. The purpose beyond keeping the process in its optimal level in the steady state conditions is also looking at ways to move from one steady state to another. Its focus is to select at each time a set of variables that allows the system under control to react more effectively when subject to disturbances. The dynamic simulation aims to achieve an optimum control system for a given process [26].

The procedure for integrating design of experiments and simulation with Aspen HYSYS to improve distillation systems is illustrated in Fig.1. The starting point in order to design and optimize a distillation system using statistical tools and modular simulators consists in choosing a case study, a distillation system to further optimize. After the selection of a case study from the literature the students need to implement and simulate the distillation system reported in literature, in order to establish the initial conditions which are the conditions described in the literature (see Fig.1).

Before establishing the initial conditions, the students need

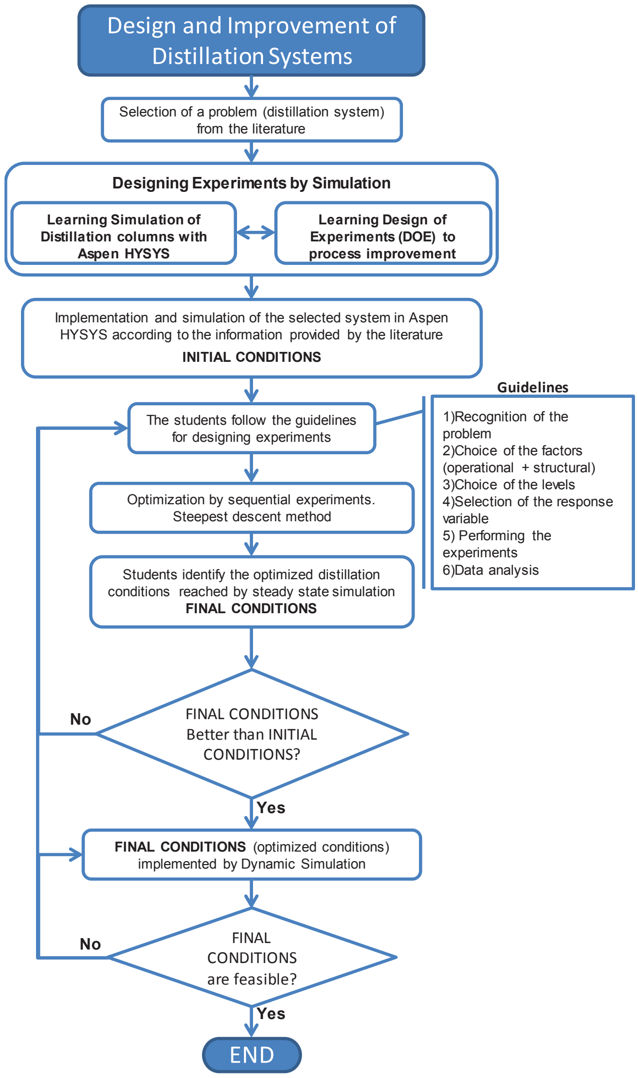


Fig. 1. Flowsheet for the design and improvement of distillation Systems integrating Aspen HYSYS and DOE.

to learn how to make simulation of distillation columns with Aspen HYSYS and how to use DOE for process improvement.

In first place the students are engaged in the learning process by the development of the activities related with the understanding of the chemical process simulation software in order to implement the case reported in the literature. This is central in learning because students do meaningful activities and think about what they are doing [27]. This is not the first time that the students are taking contact with Aspen HYSYS because at this point of the chemical engineering master course the students already worked with Aspen HYSYS in two different curricular units and so they are familiar with the basic issues. However, the problems selected from literature are complex and require some time and effort to implement. Moreover, they have a focus on novel distillation concepts from the students´ perspective with the perception of meeting the demands of a more sustainable modern society, focusing in the reduction of costs improving eco-efficiency.

After implementing in Aspen HYSYS the case study selected from the literature the students are encouraged to perform some simulations in order to further improve the distillation system object of the study following a student centred inductive approach. The main objective is to shift the focus of the responsibility of learning to the student by helping the student to develop autonomy in approaching a new problem and enhancing the skills to gather data and make informed decisions.

Concerning the design of experiments (DOE) it is the first time that the chemical engineering master students are taking contact with the techniques. In order to students be aware of the main issues of process optimization with design of experiments (DOE) the teacher recognize what is important for the students to learn before they engage an active learning approach. The elementary principles and techniques of statistical experimentation, mainly full factorial designs, fractional factorial designs, and data analysis are firstly introduced to the students with a deductive learning process with special focus placed on experimentation for process improvement.

After understanding the elementary issues of design of experiments (DOE) the students are able to look at the problem and start to plan the experiments to run. The students need to take their own decisions concerning the experiments to perform in order to identify the vital sources of variation and quantify the effects of the control variables including the interactions between the variables. The students apply the DOE approach to the particular problem they face based on several control factors and on the desired outcome response according to the guidelines described in Fig.1. The students follow the steps for planning, conducting and analysing the experiments.

After performing the experiments by simulation with Aspen HYSYS and analysing graphically and statistically, the students decide which the best levels of the control factors are in order to have the desired outcome. At the end, the conditions are compared with the starting point of the literature case study to verify the improved achievements.

After performing the steady state simulation and reaching the optimized conditions it is desirable to perform dynamic simulation. The students are motivated to implement the dynamic simulation in order to deal with disturbances.

## IV. Design and improvement of Distillation Systems – two case studies from a Portuguese Chemical Engineering Master Course

The selected problems are based on distillation since this remains the primary separation process used in industry for the separation of liquid mixtures. The challenge of both projects are to study alternatives in terms of configuration and design of distillation structures to improve the efficiency of the process and reduce energy consumption which is of growing concern nowadays.

Concerning energy requirements, separation sequences using conventional distillation columns (a single feed with two product streams, condenser and reboiler) undergo inherent inefficiency produced by the thermodynamic irreversibility during the mixing of streams at the feed, top and bottom of the column. This is inherent to any separation that involves intermediate boiling component and generalized to an N component mixture [28]. Theoretical studies developed by Petlyuk and co-workers [29] showed that this inefficiency can be improved by removing some heat exchangers (condensers and/or reboilers) and introducing thermal coupling between the columns.

Two different case studies were given to the students involving distillation processes with thermal coupling. Despite the high potential of the distillation process with thermal coupling economic benefits, a lack of reliable design methods has contributed for the low number of commercial solutions [30].

Therefore, it is still a challenging task for engineers to define near optimal design conditions for those systems in a simple and efficient manner in the initial stage of the design procedure. Several distinct configurations of thermal coupling systems can be implemented in commercial process simulators, but the challenge is to find optimal or near optimal solutions for the problem due to the large number of design variables of those systems which lead to tedious iterative simulations in order to find a proper structure.

Outlined below are two case studies following the procedure described in section 3.

### A. *Case study I*

This case consists of studying the optimal conditions for the fully thermally coupled distillation columns, FTCDC, through process simulation with Aspen HYSYS and statistical experimental design to separate a mixture of 2-methylpropan-1-ol, butan-1-ol and butan-2-ol. The student deepened his knowledge in simulation of distillation columns and also in techniques of experimental design by an active learning approach previously to the establishment of the initial conditions of the selected system.

To implement the initial conditions, the student started the design using the methodology proposed by Triantafyllou and Smith [31], using the preliminary design equations based on short-cut Fenske-Underwood-Gilliland-Kirkbride method (FUGK) to find the initial configuration for the Petlyuk column. The design was implemented in Aspen HYSYS v7.3 and for the computation of the thermodynamic properties it was used the UNIQUAC model with the binary parameters from the Aspen HYSYS database. A prefractionator followed by a product column characterizes this system. The first step of the design procedure applied the short-cut distillation method (FUGK) to obtain a first approximation for the Petlyuk structure as depicted in Fig. 2. In this step, the student was able to identify the values, work levels, for the main variables of this system and implemented a new structure consisting on an absorber, which corresponds to the prefractionator, and a distillation column. The student followed the guidelines for designing the experiments and identified the main design factors which were aggregated into two types: six structural factors and two operational factors as displayed in Table I.

Some special types of factorial designs are very useful in process development and improvement. One of such kinds are factorials of the type $2^k$ with k factors, each at two levels usually referred as low level (-1) and high level (+1) of the factor. As the number of factors in a factorial experiment grows the number of effects to estimate also grows.

In the present case the student identified a total of 8 factors and concluded that would need a total of 256 simulations (experiments) in order to perform all the combinations which would be rather time consuming. In order to reduce the number of simulations and assuming the sparsity of effects principle a fractional factorial design was selected to obtain information on the main effects and low order interactions. The fractional factorial chosen was $2_{IV}^{8-4}$. This design only requires 16 experiments reducing considerably the number of runs required for a full factorial experiment. For the fractional factorial design four generators were used, E=BCD, F=ADC, G=ABC and H=ABD. In order to interpret the results of fractional factorial designs it is necessary to take into account the alias relationships [24].

TABLE I. MAIN FACTORS AND INITIAL CONDITIONS – CASE I

| Structural factors | |
|---|---|
| NF=8 – Position of the feed stream in the prefractionator (factor A) | NTS2=51 – Number of stages in the middle section of the main column. (factor D) |
| NTP=19 – Number of stages of the prefractionator (factor B) | NS=42 – Position of the extraction of B product (middle). (factor E) |
| NTS1=15 – Number of stages in the top section of the main column. (factor C) | NTS3=8 – Number of stages in the bottom section of the main column. (factor F) |
| **Operational factors** | |
| V3=280 kgmol/h – Vapor Molar flow of the bottom interconnection stream. (factor G) | L1=240 kgmol/h – Liquid molar flow of the top interconnection stream. (factor H) |

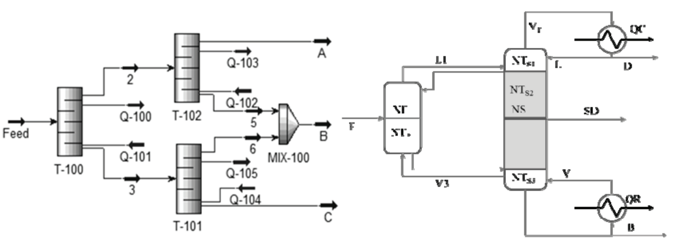


Fig. 2. Implementation of the FUGK method in Aspen HYSYS and identification of the main factors of the Petlyuk system.

For the design of experiments simulations, a variation of ± 1 stage was used for the structural factors and a variation of ± 5 kgmol/h for the operational factors in relation to the initial conditions. The response variable selected was the total cost obtained with the Aspen Economic Evaluation using the default definition [32]. With this tool, it is possible to obtain a rapid estimation of the capital and operational cost of each run. After performing the 16 experiments, the effects were estimated and a normal probability plot of the effects was built, as presented in Fig. 3a, in order to graphically judge the relevance of the factors and interactions. After ANOVA computation the student concluded that all the seven, factors and interactions, affect significantly the total cost (response variable), a result already observed graphically (see Fig. 3a) and tested with ANOVA. The conclusion was that factors A, B, G and H are significant as well as the interactions AB, BG and GH. Fig. 3b represents the plots of the AB, BG and GH interactions and A, B, G and H main effects. The plot of the interaction GH shows that the interaction is very strong, and the effect of changing H from the lower level to the higher level is dependent of the level in which factor G is settled (the interaction hide the main effects). Looking at Fig. 3b it was easy to conclude that it is better to work with factors A and B in their higher levels in order to minimize the cost. In relation to factors G and H it is better to work with factor H in the lower level an also factor G in the lower level due to the effect of the interaction GH that is stronger than the effect of the individual factors.

After performing the fractional factorial design for process characterization the next step was the process optimization in order to find the set of conditions that result in the lowest total cost. In order to optimize the student decided to use the method of steepest descent, which is a procedure for moving sequentially along the path of steepest descent that is in the direction of the minimization of the response. A second cycle of simulations were performed varying the factors considered significant. The experiments were conducted along the path of steepest descent with a full factorial design $2^4$ with the factors A, B, G, and H varying in the direction of the better level in

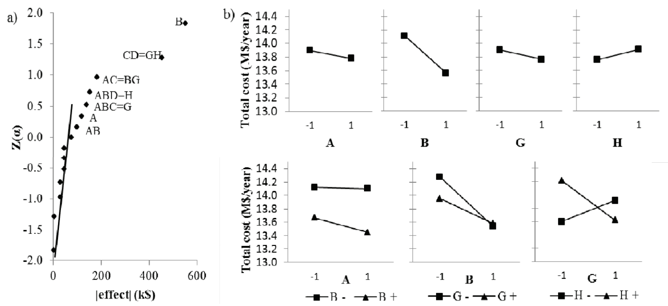


Fig. 3. Factorial fractional experiments results: a) Probability plot to identify the significant factors and interactions; b) Influence of the factors and interactions level in the total cost.

which the total cost reduces. The results of ANOVA for a level of significance of 5 % allowed to the conclusion that the factors A, B, G and H still affect the response. After that, a third designed experiment was performed in order to further move along the path of steepest descent. After the whole process of optimization done sequentially, the final conditions of the eight factors (structural and operational variables) in order to minimize the total cost are: A – NF=13; B – NTP=32; C – NTS1=15; D – NTS2=51; E – NSD=42; F – NTS3=8; G – V3=265 kgmol/h; H – L1=225 kgmol/h. The use of these conditions in the Petlyuk column allows a reduction of the total cost of 9.6 % relative to the initial conditions, as reported in the case study selected from the literature.

After perform the statistical designed experiments, the student was able to verify that the final conditions were better than the initial ones and so the next step consisted in testing the final conditions by dynamic simulation using the Aspen HYSYS in dynamic mode. This type of system is more complex than a traditional distillation column with more degrees of freedom. Taking in consideration the control objective and previous work done by Wolf and Skogestad [33] and Hwang et al. [34] it was chosen a LV configuration with SISO feedback control loops, adapted from the LV configuration used in traditional distillation columns.

The control structure implemented presents eight control loops and is represented in Fig. 4.

The control of the FTCDC system is more complex than in a traditional distillation column and the results obtained showed that the response time of the system is higher when compared with a system of two columns for the same separation, but even so possible to control. The FTCDC control can be improved using model predictive control strategies.

The student concluded that the combination of statistical tools like DOE proved to be very useful in the simulation of multicomponent separation reducing considerably the number of simulation runs to achieve an optimized solution for a particular problem. In the case of this Petlyuk column a reduction of the total cost estimation of almost 10 % was obtained with a reduced number of runs. The dynamic simulation of the optimized Petlyuk column showed the possibility of operation of this system, but it is still necessary to implement new control strategies to overcome the high response time observed with the use of model predictive control as strategy to improve the performance of these systems [35].

*B. Case study II*

This case concerns the design and optimization of an azeotropic distillation system with thermal coupling. A bioethanol dehydration process, requiring a significant amount of energy to overcome the azeotrope behaviour of ethanol-water mixture, was selected from the literature. The distillation system reported in the literature illustrates a new alternative process of intensification based on a dividing wall column (DWC) with energy saving and less equipment units when compared to the conventional azeotropic by extractive distillation configurations [36]. The student implemented by simulation with Aspen HYSYS v7.3 software this process consisting of a mixture of water and ethanol. The feed composition of ethanol is slightly lower than that of the composition of the well-known binary azeotrope formed between constituents. It was used n-pentane as mass agent to break the azeotrope obtaining a heterogeneous azeotropic distillation scheme as shown in Fig.5.

The system consists of two columns connected in reverse order. In the main column (MC) it takes place the azeotropic distillation, yielding the ternary azeotrope as a distillate and ethanol as a residue. The side stripper (SS) performs the recovery of the mass agent on the distillate stream, which is recirculated to MC. The residue stream (R1) from this column consists essentially of water. Both columns are joined by two streams, vapour (V1) and liquid (L1) in a thermally coupled distillation columns with side stripper (TCDC-SS) system.

After establishing the initial conditions of the system the student selected by hands on approach the factors to optimize namely structural and operational factors and the response variable, using the guidelines for designing experiments. Twelve factors were identified in order to optimize the system consisting of seven structural factors and five operational factors as depicted in Table II.

With 12 factors, the number of runs required to perform a full factorial design ($2^{12}$) would require 4096 simulations, which would be unfeasible, due to the huge time required. However, the objective was to obtain information on the main effects and low order interactions and so a fractional factorial design would serve the purpose with the benefit of the reduced number of simulations to perform. Thus, a fractional factorial design $2_{IV}^{(12-7)}$ was adopted, with the design generators F=ACE, G=ACD, H=ABD, J=ABE, K=CDE, L=ABCDE and M=ADE, obtaining a design matrix of 32 of experiments by

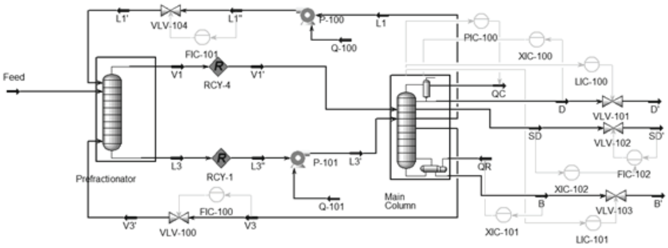


Fig. 4. Implementation of FTCDC in Aspen HYSYS with control loops.

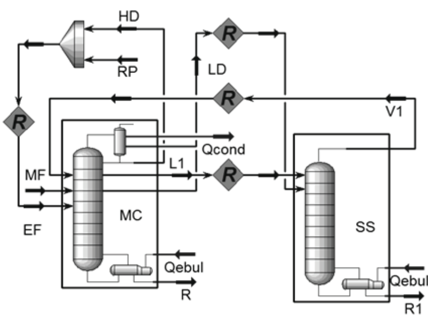


Fig. 5. Heterogeneous azeotropic distillation scheme implemented in Aspen HYSYS.

TABLE II. MAIN FACTORS AND INITIAL CONDITIONS – CASE II

| Structural factors | |
|---|---|
| The number of stages of MC - 35 (factor A) | The feed stage of the vapour stream (V1) to MC - (factor E) |
| The feed stage of the feed mixture to MC – 15 (factor B) | The number of stages of the SS – 26 (factor F) |
| The feed stage of the mass agent to MC – 15 (factor C) | The number of the feed stage of the SS – 10 (factor G) |
| The outlet side stream stage of MC – 10 (factor D) | |
| **Operational factors** | |
| The flow rate of the liquid outlet side stream (L1) from MC – 120 kgmol.h-1 (factor H) | The flow rate of n-pentane in circulation – 800 kgmol.h-1 (factor L) |
| The flow rate of vapour stream (V1) from SS recirculated to MC – 160 kgmol.h-1 (factor J) | Reflux ratio of the main column – 0.93 (factor M) |
| Main column and side-stripper top pressures – 1.2 bar (factor K) | |

simulation without replication [37].

With this design the 32 simulations were performed making changes in the structural and operating variables and the corresponding changes in the response variable (i.e. the respective total (investment plus operative) annualized costs for the several experiments, obtained through the Aspen Economic Evaluation.

To analyse the data from the fractional factorial design the analysis of variance (ANOVA) was used and the computed F ratios compared to a 5% upper critical value of F distribution concluding that factors H, J, L, M and the interactions KL, LM, JM, ADL, DL and AL are significant. After the first fractional factorial design, and once the appropriate set of factors were identified, optimization was performed in a sequential procedure with the method of steepest descent moving sequentially along the direction of the maximum decrease in the response.

Fig. 6 presents the total cost starting with the base case (i.e. initial conditions), the case study reported in the literature, by implementation of the conditions of the work [36] followed by the sequential designed experiments achieving a cost reduction of 32.1 %. The student was able to conclude the benefits of Integrating Design of Experiments and Simulation with Aspen HYSYS in order to improve Distillation Systems. With this procedure it was possible to reach a huge reduction of the cost.

After the 5$^{th}$ experiment the final conditions obtained by steady state simulation for the system TCDC-SS are presented in Table III, and correspond to the optimized conditions.

The work levels of the improved system obtained by simulation in steady state were then tested by dynamic simulation. The goal of dynamic simulation is to verify if the system meets the designed objectives ensuring the specification in terms of purity of the product of interest (ethanol). In order to make the transition from steady state to dynamic and perform the analysis of controllability it is required to perform some actions and changes concerning the process diagram created for the steady state and set an appropriate control strategy according to Aspen HYSYS - Dynamic Modelling Guide V.7.3. The control structure was defined and the

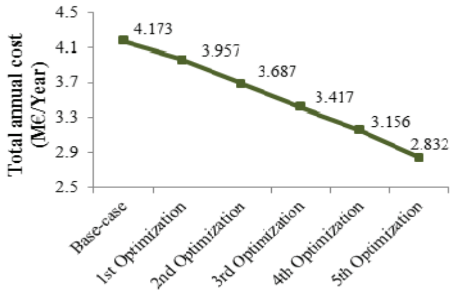


Fig. 6. Total annual cost versus optimization process for the azeotrope distillation system

TABLE III. WORK LEVELS OF THE FACTORS IN THE IMPROVED SYSTEM.

| Factor | Work levels | Factor | Work levels |
|---|---|---|---|
| A | 33 | G | 12 |
| B | 17 | H | 108 kgmol.h$^{-1}$ |
| C | 17 | J | 120 kgmol.h$^{-1}$ |
| D | 8 | K | 1.1 bar |
| E | 12 | L | 290 kgmol.h$^{-1}$ |
| F | 23 | M | 0.70 |

applicable control loops introduced. After tuning the various controllers, it was possible to obtain a stabilized system. The student also analysed the overall behaviour of the system due to the introduction of disturbances. The student was able to conclude that the use of statistical experimental design combined with process simulation proved very effective in order to drastically reduce the costs for the separation process of ethanol-water mixture through azeotropic distillation. It was also possible to implement a control structure that allowed an adequate response in relation to the introduced disturbances, proving that the final conditions are feasible [38].

## V. EVALUATION OF THE PROJECTS AND IMPACT ON STUDENTS LEARNING

### *A. Evaluation of the projects*

A concluded master's thesis is evidence that the students from the master program are fully qualified engineers who can add value to the success of the organization for which they will be working.

The two projects presented have been evaluated according to the rules of the master project. It must be clear from the master´s work that the students are able of dealing with a concrete subject or problem of considerable magnitude and capable of offering a solution to that problem by using their knowledge and ability.

Each final project was performed by one student according to the rules of the master project and each student presented a written report, which was evaluated by a jury and also an oral presentation in order to defend the project in a public presentation. The key elements that the students had to demonstrate in their master project include: a) Were the objectives clearly stated?; b) Was the problem well defined?; c) Were the design, modelling and analysis clearly understood?;

d) Are the results technically and economically feasible?; e) Are the conclusions effective?; f) Was the content well organized?; g) Was there an appropriate use of engineering tools necessary to engineering practice?; h) Was the message clearly delivered?

Each written report was evaluated by a jury with respect to the content and quality of the project. The jury evaluated the extent to which the research is innovative (i.e. contribution to a concrete system design new or improved one, the creativity and independence of the student in the introduction of new or improved concepts), the application of the correct research and methodologies, the extent to which the research proposal has been met, the relevance of the project (i.e the applicability in practice, ability to put the research in context).

The independence and professional skills of the students were evaluated (e.g. independence, assimilation of feedback, communication skills, cooperation) as well as the students attitude to reinforce his/her individual development, the commitment and enthusiasm with the project and the reflection upon his/her own work.

Each student's oral presentation has also been evaluated by the corresponding examination panel concerning the ability to communicate effectively (e.g. if the message is coming across, if the student has a captivating way of presenting concerning verbal capabilities as well as posture), insight in subject matter and coherence between different parts of the project (i.e. the knowledge concerning contemporary analytical, computational and experimental practices, the know how in chemical process simulation software, and the skills in statistical experimental design, planning, conducting the experiments and the statistical analyses of the results).

The two examples of master projects described provided the students an opportunity to demonstrate acquired competences and skills concerning design, analysis, and control of advanced chemical processes. This includes abilities in requirements such as: a) Familiarity with statistics in real application environment; b) Competences in experimental design, data collection and data analysis; c) An ability to work professionally in chemical systems areas including the design analysis and improvement of such systems; d) Skills in the use of computational tools and e) Independent thinking, creativity and ability to tackle advanced chemical engineering problems in real contexts.

### *B. Impact of the projects on students learning*

The methodology used in both projects had a great impact on both students especially due to the hands on approach in statistical experimental design techniques and optimization.

At the beginning of the project the students were a little bit worried about the use of Design of Experiments (DOE), mainly the use of factorial and fractional factorial designs in the context of simulation with Aspen HYSYS tools. The main reasons advocated by the students was the fact that they do not anticipate using this kind of tools after graduation specially because they were not used to deal with statistical tools in industrial or quasi industrial settings. This came on agreement with the work developed by other authors where they refer almost the same fears of the students [39].

The work had a great impact on student´s efforts to accomplish the tasks. According to students´ opinions the methodology followed was very helpful in order to increase their ability to work independently.

The students exhibited a good attitude during the progress meetings and revealed active reflection upon their own work. The work also had a great impact in raising the communication ability of the students because the students felt stress-free in reporting the progress on intermediate steps.

The students found the methodology very useful as reported by the comments they made during the project and also during the discussion of the project. The comments were very positive and some of their remarks are summarized below:

"I found the use of statistical design of experiments very challenging because I had no previous experience on using these type of statistical tools during my course"

"I found the project very challenging mainly due to the objective of optimizing a system that was already published in literature and I was afraid that there was not much space in order to further improve the system, which came to be a wrong thought".

"The implementation of the system to optimize was useful and allowed to apply my Aspen HYSYS skills"

"I learn by hands on approach how to plan and perform the experiments and I think that DOE will be useful in my future profession"

"I learned to model dynamic processes and this is likely to have much interest in the future as an engineer".

"I found very challenging looking for the information needed in order to solve the problem in hands"

"I improved my skills in simulation with Aspen HYSYS concerning the conceptual design of distillation schemes for the separation of chemical mixtures"

The comments of the students were very informative and in line with the objectives of the project. The students found very enriching using statistical experimental design techniques along with simulation using Aspen HYSYS by hands on approach procedure and they were able to plan and perform the experiments using the right research and design methodologies.

## VI. Conclusions

The procedure described in this paper was a good educational experience for both students because they had the opportunity to perform meaningful activities and think by themselves in face of real problems. This kind of inductive teaching and learning showed very enriching because it imposed more responsibility to the students for their own learning. In fact, students build their own thinking, discussed questions with the teachers and solved problems by an active learning process. The teachers guided the process without controlling the students´ choices and that is an important issue to have into consideration. This is in accordance with de Graaff and

Mierson [40] and Margetson [41] which refer that the role of the teacher is very challenging involving more than just subject knowledge. The students followed the orientations of the teachers but they were responsible for their decisions meaning that they decided how to implement the systems and the methods to use in order to optimize the systems. Students recognised the problem under study and were able to realize the complexity of the systems. The optimization of complex and real problems help them to develop content knowledge as well as problem solving, reasoning and self-assessment skills. The students realized that they were also acquiring the skills needed to be successful in the field because they had a clear picture of the role that chemical engineers may have in improving the performance of the processes and recognized the relevance of undertaking engineering activities in a way that contributes to sustainable development. Students also understood that computer software process simulation tools are essential for providing fast and, as much as possible, accurate solutions. Aspen HYSYS software for process simulation enabled simple and quick conceptual design of distillation structures in order to make separations of chemical mixtures being crucial in the design, analysis and evaluation of the processes. Both students found very enriching the fact of being able to identify the factors to optimize and select the response variable to measure and also to plan and develop the simulation runs and statistical analysis by themselves, evaluating and deciding which runs to perform in order to optimize the examples selected from the literature. The students found very motivational taking their own decisions concerning the procedure for moving sequentially along the direction of the costs minimization. The students also found very useful the knowledge they got in factorial and fractional factorial designs particularly the usefulness of these designs for screening the variables in the distillation system and determining those that are more important. They learned how to analyse the data from factorial designs, namely by analysis of variance (ANOVA) and also how residuals are used for model adequacy checking for factorial designs.

The students felt the approach very useful to enrich their technical knowledge in relevant areas of chemical engineering increasing their expertise in tools which are widely-used across the industry to model steady-state or dynamic processes and to perform studies in order to optimize the systems. They also found that the work developed helped them to be conscious about the information they need to know to solve the problem in hands, about the strategies to use in order to optimize the systems and above all allowed them to see the impact of the engineering solutions they reached.

From the teachers perspective it was very important that students could learn by a hands on approach procedure how to improve a process with designed experiments. The simulation of distillation systems with tools, like Aspen HYSYS that the students already knew from some curricular units taught in the course was a starting point to deal with statistical experimental design in order to optimize a system published in the literature. The great advantage of the methodology is that the students could easily understand important topics of statistical experimental design including the analysis of factorial experimental designs and the use of graphical methods such as the interaction graphs so useful in the interpretation of the results.

The use of such tools associated with real cases allows students to see how useful statistics can be in their late careers in industry. The students improve their skills in problem analysis being able to convert an engineering problem into statistical terms from which appropriate solutions can be chosen.

From the teachers viewpoint it is advantageous to integrate DOE with process simulation tools because it can overcome some issues related with available financial resources to explain and training this type of statistical techniques. Globally the methodology was very helpful to promote self-directed learning and the adoption of a deep approach to learning where the students assumed a high level of responsibility for their own learning.

In both projects the students improved a specific distillation system using chemical process simulation and designed experiments. Each project was a good opportunity that students fully used to act as an engineer applying their scientific and technical knowledge to successfully optimize a distillation system.

This kind of projects puts the students in connection with industrially relevant separation processes, namely distillation that is still nowadays the main separation process used in chemical industries for separation of liquid mixtures.

Due to the success of this type of procedure it is worth to apply to other type of systems and also to other type of chemical operations meaning that the work will have a potential impact on a wider public because this type of procedure using DOE by simulation is extensible to other type of processes and simulators and can be placed in a wider educational context.

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