During their fourth year of undergraduate studies, chemical engineering students at the Instituto Superior de Engenharia de Coimbra (ISEC), Portugal, take a full laboratory course in unit operations and process control. The topics covered include evaporation, distillation, absorption in packed columns, solid-liquid extraction, drying, and the control of variables often found in industrial units (e.g., pressure, flow, level, and temperature) employing laboratory units or bench-scale kits. The course’s basic aim is the practical demonstration of theoretical concepts taught in courses on process separation, chemical thermodynamics, and process dynamics at laboratory scale. It also provides students with experience in operating and controlling complex units. Regarding the work on distillation, the students are asked to validate the steady-state behavior of a laboratory unit used for separating an azeotropic mixture of aniline and water. The interest in this binary system arose from an intensive research program carried out in the chemical engineering department of ISEC in collaboration with a Portuguese company that produces aniline—with the aim of optimizing the aniline production section. In addition, this work also aims to validate knowledge relating to the distillation of heterogeneous azeotropic mixtures, of which the aniline-water system is a simple and easily handled example.

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Validating
THE EQUILIBRIUM STAGE MODEL
for an Azeotropic System in a Laboratorial Distillation Column

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The conceptual basis used to describe the phenomena involved in a distillation column is the equilibrium stage model, which assumes thermodynamic equilibrium between
the mismatch of theoretical assumptions and real behavior, the equilibrium stage model is still used to represent the operation of distillation columns phenomenologically, and several research groups have presented data for three-phase distillation experiments to validate it. Thus, this work aims simultaneously to enable students to gain experience in operating and analyzing a distillation procedure, to use vapor-liquid equilibrium (VLE) prediction methods, and to contribute to the research community’s efforts to validate the equilibrium stage model for the aniline-water system.

Three four-hour sessions are required to complete the work. The first is devoted to a review of the basic theory behind mixture thermodynamics and methods for predicting activity coefficients for VLE, analysis of the column layout, and understanding its operation and control. Between the first and second sessions, the students develop an Excel workbook to obtain the VLE prediction for the aniline-water system at atmospheric pressure. Each group of three or four students is asked to use a different prediction method for the activity coefficients among UNIFAC, UNIQUAC, two-constant Margules, and van Laar, then to compare the results with experimental data published in the literature for the aniline-water system at atmospheric pressure. 

During the second session, the students carry out the experimental work with the distillation column, running it until steady-state conditions are reached. Next, they use Aspen Plus v. 11.1 to compute the unit steady state with an equilibrium stage-model-based module. Finally, in the third session, the results are presented, compared with those given by simulation, and discussed.

EXPERIMENTAL EQUIPMENT AND SAMPLING

Aniline, even in low concentrations, is a fairly toxic aromatic hydrocarbon. Its handling therefore requires students take some safety precautions, that is, to wear protective clothing, gloves, and eye/face protection to avoid skin contact. Furthermore, the work is carried out in a well-ventilated area to reduce the toxicity risk.

Figure 1. Schematic diagram of laboratory unit used for experiments.
Figure 2. Schematic representation of column trays.  

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Aqueous Phase</th>
<th>Organic Phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>999</td>
<td>1023</td>
</tr>
<tr>
<td>30</td>
<td>997</td>
<td>1014</td>
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<td>1006</td>
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<td>987</td>
<td>989</td>
</tr>
<tr>
<td>70</td>
<td>982</td>
<td>982</td>
</tr>
</tbody>
</table>

Table 1: Density of Aniline-Water System for Different Temperatures [8, Appendix G]

To overcome this drawback, instead of reflux ratio adjustment, an on-off control scheme based on the boiling point limit in the upper tray of the column is employed, with the head temperature measured through a resistance thermometer (Figure 1). Whenever it exceeds the value selected at the control unit (98.7 °C—the azeotropic temperature), the distillate flow rate is automatically interrupted, and the condensate starts to flow back to the column. Consequently, the temperature of the upper tray starts to fall, and when it reaches the preselected value the distillate starts to be collected again. This control scheme leads to small changes in the position of the solenoid valve, which is open most of the time.

The feed stream is preheated to 99.3 °C before entering the column in liquid state at atmospheric pressure. The feed stream is sampled for the quantitative determination of aniline concentration in the mixture.

When the steady state is reached, the students collect the distillate and bottom products in graduated receivers for a period of 90 minutes. At the end, the distillate is transferred to a separatory funnel, and after the separation of the two phases, the volume of each layer is measured in graduated cylinders.

The aniline concentrations in the aqueous phase of distillate, in the bottom product, and in the feed sample are quantitatively determined by spectrophotometry, after appropriate dilution. The absorbance of the solutions is measured at 279.5 nm in a UV-Vis spectrophotometer unit after setting the calibration line. The quantitative analysis of water concentration in the organic phase of the distillate is determined by titration with Karl Fisher reagent.

For the range of flows used in the experiments, the column manufacturer indicates that the Murphree efficiency is 92%. The experimental confirmation of this value is not performed exactly because of the column configuration, which does not allow the extraction of liquid samples in consecutive trays.

**MODEL VALIDATION**

Aspen Plus v. 11.1 is used to validate the equilibrium stage model; the steady-state flows, compositions, and temperatures of the upper tray and bottom stream result-
ing from the model solution are compared with laboratory data. The Radfrac module is chosen to describe the column unit since it is based on the rigorous solution of the equilibrium stage model for multistage vapor-liquid fractionation, steady-state operations.\cite{9, 10} The model consists of a set of nonlinear algebraic equations, comprising the material balance (M) and thermodynamic equilibrium relation (E) for each component and tray, and the summation of mole fractions (S) and enthalpy balance (H) for each tray, generally called MESH equations.\cite{11}

This system can be augmented with the trays' hydraulic relations and pressure-drop profiles across the column when the unit geometry is known. The broader generality of the Newton-Raphson algorithm led this to be chosen to solve Radfrac module in rating mode. The Newton-Raphson algorithm implemented is based on the classic Naphitali and Sandholm algorithm.\cite{12} First, the number of equations and variables resulting from unit modeling is reduced through the condensation of mole fractions, liquid, and vapor flows into new variables representing component molar flows. Next, the complete set of variables is ordered, and the resulting algebraic equation system solved iteratively by employing a Newton-type algorithm. The convergence is checked after each iteration by comparing the sum of squares of all variables, conveniently weighted by scale factors, with a tolerance defined as a function of the number of degrees of freedom the system involves and inlet flows.

An azeotropic convergence algorithm is chosen to handle the current binary system that forms a minimum-boiling azeotrope in the region of low aniline concentrations. The operating conditions, including the molar flow of distillate stream, the heat consumed in reboiler, the stages at which streams enter/leave the unit, the pressure-drop profile across the column, additional information regarding the condenser operation, and the characterization of the second liquid phase—formed essentially by aniline—are introduced into Aspen Plus. Molar flows are set equal to the experimental steady-state values, and the pressure drop is disregarded due to the small flows involved in the operation. The characteristics of the feed stream, including its temperature, pressure, and molar composition, are also entered into Aspen Plus. It is considered that there is no sub-cooling in the condenser and the Murphree stage efficiency is set to 92%.

The Appendix at the end of this paper presents the Aspen Plus Input Summary file for a successfully converged model.

RESULTS

The first step in analyzing the results is to compare the VLE data calculated by the Aspen Properties module with the prediction obtained by students. Next, both must be validated with experimental data. The diagram in Figure 4 shows the agreement between the VLE data predicted using the UNIFAC method and the experimental data published in the literature.\cite{8, Appendix G} The system presents a heterogeneous azeotrope at 98.7 °C and 0.044 of aniline mole fraction, where three phases are in equilibrium: a vapor phase and two liquid phases [an organic phase with 30.3% (mole/mole) water, and an aqueous phase with 98.6% water].

This happens because the vapor-liquid envelope overlaps the liquid-liquid envelope, as illustrated in Figure 4. This task allows students to understand that for heterogeneous azeotropes the vapor formed during boiling has the same composition as the overall liquid, but the three phases in equilibrium have distinct compositions, contrary to what

![Figure 4. Comparison of experimental data from literature vs. VLE data determined through UNIFAC for aniline-water system.](image-url)
happens for homogeneous azeotropes, where the liquid and vapor formed have the same composition.

The VLE prediction methods using UNIQUAC, two-constant Margules, and van Laar activity-coefficient models require binary parameters for the aniline-water system, which students estimate by employing the following procedure and theoretical basis.

For a binary system containing two liquid phases and one vapor phase in equilibrium, the fugacities of each compound in each of the phases are equal. That is:

\[
\gamma_{\text{org}}^{\text{org}} P_{\text{org}} x_{\text{org}}^{\text{org}} = \gamma_{\text{org}}^{\text{aq}} P_{\text{org}} x_{\text{org}}^{\text{aq}} = P y_2
\]

and

\[
\gamma_{\text{org}}^{\text{org}} P_{\text{org}} x_{\text{org}}^{\text{org}} = \gamma_{\text{org}}^{\text{aq}} P_{\text{org}} x_{\text{org}}^{\text{aq}} = P y_2
\]

when the vapor phase behaves like an ideal gas mixture. The expressions \(\gamma_i^{\text{org}}\) and \(\gamma_i^{\text{aq}}\) represent the activity coefficients of component \(i\) in the organic and aqueous phases, respectively; \(x_i^{\text{org}}\) and \(x_i^{\text{aq}}\) are the molar fractions of component \(i\) in the organic and aqueous phases, respectively; \(P\) is the vapor pressure of the pure component \(i\) at temperature \(T\); \(P\) is the pressure; and \(y_2\) is the mole fraction of component \(i\) in the vapor phase. The subscript \(s\) stands for saturated liquid phases.

According to the phase rule, a binary system with three phases in equilibrium has just one degree of freedom, which means that by fixing the pressure (atmospheric pressure) the system becomes determined. Setting the activity coefficient model, the functional forms of \(\gamma_i^{\text{org}}\) and \(\gamma_i^{\text{aq}}\) can be explicitly written, and the preceding equations lead to

\[
\gamma_{\text{org}}^{\text{org}} \left(A_{\text{a,w}} x_{\text{org}}^{\text{org}} A_{\text{a,a}} x_{\text{org}}^{\text{org}}\right) x_{\text{org}}^{\text{org}} = \gamma_{\text{org}}^{\text{aq}} \left(A_{\text{a,w}} x_{\text{org}}^{\text{aq}} A_{\text{a,a}} x_{\text{org}}^{\text{aq}}\right) x_{\text{org}}^{\text{aq}}
\]

and

\[
\gamma_{\text{org}}^{\text{org}} \left(A_{\text{a,w}} x_{\text{org}}^{\text{org}} A_{\text{a,a}} x_{\text{org}}^{\text{org}}\right) x_{\text{org}}^{\text{org}} = \gamma_{\text{org}}^{\text{aq}} \left(A_{\text{a,w}} x_{\text{org}}^{\text{aq}} A_{\text{a,a}} x_{\text{org}}^{\text{aq}}\right) x_{\text{org}}^{\text{aq}}
\]

where \(A_{\text{a,w}}\) and \(A_{\text{a,a}}\) are the binary interaction parameters of the model chosen for the aniline-water system. When the liquid-liquid equilibrium data is available, the fractions \(x_{\text{org}}^{\text{org}}\), \(x_{\text{org}}^{\text{org}}\), \(x_{\text{org}}^{\text{org}}\), and \(x_{\text{org}}^{\text{org}}\) can be used to evaluate the two parameters \(A_{\text{a,w}}\) and \(A_{\text{a,a}}\). Solubility data of aniline in water and of water in aniline for a temperature range of 20 °C to 100 °C can be found in the reference [8, Appendix G] thus allowing students to estimate mutual solubility values at the azeotrope temperature (98.7 °C). The parameters obtained are used to calculate activity coefficients for subsequent vapor-liquid equilibrium calculations in the regions of \(0 < x_{\text{org}}^{\text{org}} < 1\), and \(x_{\text{org}}^{\text{org}} < x_{\text{org}}^{\text{org}} < 0 < 1\). Table 2 lists the parameters obtained by the students for the UNIQUAC, two-constant Margules, and van Laar activity coefficient models.

The results of the Radfrac module are compared with experimental results in Table 3, showing the agreement

### Table 2

**Values of Binary Interaction Parameters for UNIQUAC, Two Constant Margules, and van Laar VLE Prediction Models**

<table>
<thead>
<tr>
<th>model</th>
<th>(A_{\text{a,a}}) (J mol(^{-1}))</th>
<th>(A_{\text{a,w}}) (J mol(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNIQUAC</td>
<td>1524</td>
<td>575</td>
</tr>
<tr>
<td>two-constant Margules</td>
<td>6247</td>
<td>5836</td>
</tr>
<tr>
<td>van Laar</td>
<td>13164</td>
<td>4798</td>
</tr>
</tbody>
</table>

### Table 3

**Comparison of Experimental Data with Model Predictions**

**Operating Conditions**

**Feed:**
- Flow: 2.623 l/h = 2.619 kg/h
- Molar flow: 132.22 mol/h (100% liquid)
- Temperature: 99.3 °C
- Pressure: 1 atm
- Aniline mole fraction: 4.53×10\(^{-3}\)

**Distillate:**
- Molar flow: 17.72 mol/h
- Heat duty: 335 W

**Reboiler:**
- Temperature: 98.98 °C
- Aniline mole fraction: 0.0338
- Reflux ratio: 0.645

**Laboratory data**

**Distillate:**
- Temperature: 98.7 °C
- Aniline mole fraction: 0.0353

**Bottom product:**
- Molar flow: 114.63 mol/h
- Temperature: 101 °C
- Aniline mole fraction: 1.14×10\(^{-6}\)

**RadFrac results**

**Distillate:**
- Temperature: 98.98 °C
- Aniline mole fraction: 0.0338
- Reflux ratio: 0.645

**Bottom product:**
- Molar flow: 114.50 mol/h
- Temperature: 100.02 °C
- Aniline mole fraction: 1.16×10\(^{-6}\)
between the model’s prediction and experimental data, thus validating the equilibrium stage model for the aniline-water system. Once the heat duty in the reboiler is introduced into the model, in rating mode, it is able to determine the reflux ratio, which just represents an average value since, with the control strategy implemented, it varies discretely between 0 and $\infty$, depending on the valve position. Indeed, such a value represents a possible set-point value, if the control scheme was based on the reflux ratio.

Using the Profiles form, it is possible to view the results from Radfrac as compositions, temperatures, and flow rates for each column tray. Figure 5 shows the profiles of aniline composition in both liquid and vapor phases across the column. As expected, the aniline concentration in both liquid and vapor streams increases from the bottom to the top of the column, with the aniline mole fraction in the vapor always being greater than that in the liquid since the feed stream is located to the right of the azeotropic point of the VLE diagram (see Figure 4). The composition profiles provide strong evidence that the number of trays is over-projected for the experimental conditions tested. Indeed, in some of the stages the enrichment of vapor phase in aniline is quite small, thus leading to the conclusion that the column can successfully separate a higher flow of feed stream (with a similar composition) with the same efficiency, provided the heat supplied to the reboiler increases and flooding does not occur.

Figure 6 shows that the liquid and vapor flows across the column are approximately constant in the enrichment and stripping zones. Since stage 11 (12 in Figure 6) is the feed stage and the state of the stream is saturated liquid, the liquid flow rate in the stripping zone is increased by an amount equal to the feed flow rate. These conditions enable the McCabe-Thiele graphical construction\textsuperscript{[13]} to be used to estimate the number of theoretical trays required to perform the separation. Typical values achieved by students are about 14 trays plus the condenser and the reboiler.
CONCLUSIONS

Operating a laboratory distillation column is a good experiment for demonstrating the application of some concepts of unit operations, vapor-liquid equilibrium prediction, and process simulation. The experiment described in this paper embraces a wide range of topics including process control, chemical analysis, and numerical methods for handling rigorous distillation models. It also enables students to gain experience in operating and controlling a distillation unit. Moreover, the results provide the research community with sufficient evidence to support the validation of the equilibrium stage model for the heterogeneous azeotropic system formed by aniline and water. The results show that the column is too large for the experimental conditions tested, and additional knowledge regarding its behavior can be acquired if the inlet flow is increased and digital temperature meters are installed in each tray to validate the temperature profile. But the experiment might be successfully applied to other azeotropic systems, such as a benzene-mononitrobenzene-water mixture.

REFERENCES


APPENDIX

AspenPlus Input Summary file

DYNAPLUS
DPLUS RESULTS=ON
TITLE 'Coluna do Isec'
IN-UNITS SI
DEF-STREAMS CONVEN ALL
SIM-OPTIONS
IN-UNITS ENG
SIM-OPTIONS NPHASE=3 ATM-PRES=1. <atm>
PARADIGM=EO
ACCOUNT-INFO USER-NAME= “BELMIRO DUARTE”
DATABANKS PURE11 / AQUEOUS / SOLIDS / INORGANIC & NOASPENPCD
PROP-SOURCES PURE11 / AQUEOUS / SOLIDS / INORGANIC
COMPONENTS
WATER H2O /
ANILINE C6H7N-1

FLOWSHEET
BLOCK C1 IN=FEED OUT=DESTIL RESID
PROPERTIES UNIFAC
PROPERTIES NRTL / UNIQUAC
PROP-DATA NRTL-1
IN-UNITS SI
PROP-LIST NRTL
BPV AL WATER ANILINE 2.238300000 362.5433000 .3000000000
0.0 & 0.0 0.0 372.1500000 441.1500000
BPV AL ANILINE WATER -.8969000000 509.3646000 .3000000000
& 0.0 0.0 0.0 372.1500000 441.1500000
PROP-DATA UNIQ-1
IN-UNITS SI
PROP-LIST UNIQ
BPV AL WATER ANILINE .6554000000 -168.0642000 0.0 0.0 & 372.1500000 441.1500000
BPV AL ANILINE WATER -.4676000000 -172.2809000 0.0 0.0 & 372.1500000 441.1500000
STREAM FEED
SUBSTREAM MIXED TEMP=99.3 <C> PRES=1 <atm> & MASS-FLOW=2.427 <kg/hr> MAXIT=100
MASS-FRAC WATER 0.977 / ANILINE 0.023

BLOCK C1 RADFRAC
PARAM NSTAGE=32 ALGORITHM=STANDARD EFF=MURPHREE &
INIT-OPTION=STANDARD MAXOL=100 TOLOL=0.0001
JMETH=INIT &
LL-METH=GIBBS NPHASE=2 DAMPING=NONE
COL-CONFIG CONDENSER=TOTAL REBOILER=KETTLE
FEEDS FEED 12 ON-STAGE
PRODUCTS RESID 32 L / DESTIL 1 L
P-SPEC 1 1. <atm> / 2 1. <atm>
COL-SPECS QN=335. MOLE-D=17.72 <mol/hr>
14
SC-REFLUX OPTION=0
STAGE-EFF 1 0.92 / 2 0.92 / 3 0.92 / 4 0.92 / 5 & 0.92 / 6 0.92 / 7 0.92 / 8 0.92 / 9 0.92 / 10 &
0.92 / 11 0.92 / 12 0.92 / 13 0.92 / 14 0.92 / &
15 0.92 / 16 0.92 / 17 0.92 / 18 0.92 / 19 0.92 / &
20 0.92 / 21 0.92 / 22 0.92 / 23 0.92 / 24 0.92 / &
25 0.92 / 26 0.92 / 27 0.92 / 28 0.92 / 29 0.92 / &
30 0.92 / 31 0.92 / 32 0.92
T-EST 1 371.1 / 2 372.3 / 3 372.6 / 4 372.6 / 5 &
372.6 / 6 372.6 / 7 372.6 / 8 372.6 / 9 372.6 / &
10 372.6 / 11 372.6 / 12 372.6 / 13 372.8 / 14 &
372.9 / 15 373. / 16 373. / 17 373.1 / 18 373.1 / &
19 373.1 / 20 373.1 / 21 373.1 / 22 373.2 / 23 &
373.2 / 24 373.2 / 25 373.2 / 26 373.2 / 27 &
373.2 / 28 373.2 / 29 373.2 / 30 373.2 / 31 &
373.2
L-EST 1 3.938E-006 / 2 3.98E-006 / 3 3.984E-006 / 4 &
3.985E-006 / 5 3.985E-006 / 6 3.985E-006 / 7 &
3.985E-006 / 11 3.985E-006 / 12 4.364E-005 / 13 &
4.366E-005 / 14 4.367E-005 / 15 4.368E-005 / 16 &
4.369E-005 / 17 4.37E-005 / 18 4.37E-005 / 19 &
4.37E-005 / 20 4.371E-005 / 21 4.371E-005 / 22 &
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4.371E-005 / 29 4.371E-005 / 30 4.371E-005 / 31 &
4.371E-005
V-EST 1 0. / 2 8.86E-006 / 3 8.902E-006 / 4 &
8.907E-006 / 5 8.907E-006 / 6 8.907E-006 / 7 &
8.907E-006 / 8 8.907E-006 / 9 8.907E-006 / 10 &
8.907E-006 / 11 8.907E-006 / 12 8.907E-006 / 13 &
8.927E-006 / 14 8.945E-006 / 15 8.939E-006 / 16 &
8.971E-006 / 17 8.986E-006 / 18 8.986E-006 / 19 &
8.999E-006 / 26 8.999E-006 / 27 9E-006 / 28 9E-006 / &
29 9E-006 / 30 9E-006 / 31 9E-006
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EO-CONV-OPTI
SENSITIVITY S-1
DEFINE Z1 BLOCK-VAR BLOCK=C1 VARIABLE=RR
SENTENCE=RESULTS
DEFINE Z2 BLOCK-VAR BLOCK=C1 VARIABLE=VRATE &
SENTENCE=PROFILE ID=2
TABULATE 1 “Z1”
TABULATE 2 “Z2”
VARY BLOCK-VAR BLOCK=C1 VARIABLE=QN SENTENCE=COL-
SPECS
RANGE LOWER="250” UPPER="350” INCR="10”
SENSITIVITY S-2
DEFINE Z1 BLOCK-VAR BLOCK=C1 VARIABLE=RR
SENTENCE=RESULTS
DEFINE Z2 BLOCK-VAR BLOCK=C1 VARIABLE=VRATE &
SENTENCE=PROFILE ID=2
TABULATE 1 “Z1”
TABULATE 2 “Z2”
VARY MASS-FLOW STREAM=FEED SUBSTREAM=MIXED
COMPONENT=ANILINE
RANGE LOWER="5.0E-6” UPPER="4.0E-5” INCR="2.5E-6”
CONV-OPTIONS
PARAM TEAR-METHOD=NEWTON SPEC-LOOP=INSIDE
STREAM-REPOR MOLEFLOW MASSFLOW
PROPERTY-REP PCES NOPARAM-PLUS ☐