Process design and simulation strategies for reactive distillation column

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ABSTRACT

In this paper general process design procedure and simulation strategies for reactive distillation (RD) are presented on results are presented. The experimental results obtained from continuous RD column from our previous work are compared with simulation results predicted by three equilibrium stage models of different depths using Aspen plus software. Three types of equilibrium stage models with different complexity are developed, parameterized and compared in simulations in this work. These models are based on the stage concept and use the assumption of vapor–liquid equilibrium on each stage model. Reaction rates for Pseudohomogenous model calculated by our laboratory batch reactor are used for simulation to yield a steady state solution. Finally the VLE data from the databank of Aspen plus is also used to simulate the RD column separately to observe how the results vary with the experimental data and the Aspen plus databank data. It is observed that there is a very good agreement between the results from the Aspen plus databank and the actual column.

Introduction

Reactive Separation Processes (RSP's) combine the unit operations of reaction and separation into a single, simultaneous operation. It provides an important synergistic effect and brings about several advantages. This fusion of reaction and separation into one combined operation brings simplicity and novelty to the process flow sheet. This technology has been proved to have conclusive advantages over conventional technologies in terms of their energy; capital cost reductions and increased reaction efficiency.[1,2] RSP's are popular for the cost savings in investment and operation garnered on successful scale-up to commercial operations. These technologies are gaining importance due to economic opportunities which have caused the emergence of new industries using new separation and process technologies, to give better efficiencies of operation and quantification of product demanded by food, pharmaceuticals, polymer, and electronic industries. Due to changes in the availability and pricing of the key resources, and growing concern for protecting our environment, these technologies are also being seen as front-runner in the field of industrial separations.[3]

Reactive distillation (RD) is an attractive and promising process, which has been established in a variety of successful commercial applications. Compared with the conventional process of carrying out reaction and separation sequentially, the *in-situ* removal of products formed in the reaction zone of a RD column can favorably influence conversion and selectivity, especially of equilibrium limited reactions. Together with a significantly reduced effort in the separation train, this can result in major energy as well as capital cost savings. [4]

However, there are feasibility constraints resulting both from thermodynamics and chemistry. [5 In addition to this, the process intensification leads to higher complexity. This holds especially true for the case of heterogeneously catalyzed RD with its complex interaction between vapor, liquid and immobilized solid catalyst. [5, 6]

Simulation Work

The procedure for the development of RD processes applied here can be divided into four steps. In the first step, the thermodynamic properties of the system are investigated to obtain a reliable description of liquid-phase nonideality that can be used for a preliminary feasibility analysis. Then, the reaction kinetics is investigated at conditions that are expected for the RD column. These results can be combined, and a simulation environment based on an equilibrium stage model can be employed. Experiments in a RD column on a laboratory scale are carried out subsequently to verify the simulation. The simulation studies can be used to identify the role of important design parameters and to propose an optimal setup. Therefore first results of simulations are presented and then simulation results obtained from Aspen plus are used to validate three models developed viz equilibrium stage Model # A, Model # B and Model # C with experimental results obtained in RD column.

This work considers the production of iso-amyl acetate by RD. The paper is focused on identifying suitable process conditions and limitations for the synthesis of iso-amyl acetate. Simulation is used with equilibrium-stage model to identify optimum processing strategies.

Adjustment of the design variables is performed to iteratively check the column performance. In this paper, the simulation results for RD of iso amyl alcohol and acetic acid to synthesize iso amyl acetate are presented and discussed in a systematic manner. A tiered approach is used in the modeling efforts. The feed flow rates employed for these simulations are ranges from 0.0175 kmol/hr to 0.07 kmol/hr (1 lph to 4 lph).[7] . The comparisons are based on the obtained temperature and composition profiles. The system is first modeled considering physical and chemical equilibrium (Model #A). Physical equilibrium with a kinetically controlled reaction is incorporated in Model # B (without decanter) and Model # C. (with decanter). Finally the feasible design is obtained using the equilibrium stage model # C.

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Assumptions regarding Column Configuration and Models

For reactive separation processes, modeling is required to describe and predict the reaction and separation that occur simultaneously. To study RD esterification for present system, reliable models are essential in order to reduce the development time and expensive experimental investigations. In this respect, traditional equilibrium-based models are used. A number of assumptions concerning column configuration are used for the equilibrium model employed in this work. The distillation column is assumed to contain N stages/segments; the condenser is stage/segment 1 and the reboiler is stage/segment N. A total condenser is used. No reaction takes place in the condenser or in the reboiler. The chemical reaction is assumed to be a pseudo-homogeneous reaction that occurs in the liquid phase, with the reaction rate a function of temperature, composition and pressure.

The equilibrium model in this work consists of the conventional MESH equations with implicit assumptions that are detailed here. Physical equilibrium is achieved on each stage. The liquid phase is well mixed. Pressure and temperature are assumed constant at each stage and are uniform throughout the liquid and vapor phases present. However, both pressure and temperature can vary from one stage to another stage. In order to reduce the experimental work, reliable and accurate process simulation tools can be used for obtaining useful information needed for the design. In the present study, the simulation is carried out using equilibrium stage model, RADFRAC available in Aspen Plus software package.(Version 7.0, 2007, Aspen Tech, Inc.) [8] RADFRAC module in the steady-state simulator Aspen Plus (Version 2007) is based upon a rigorous equilibrium stage model for solving the mass balance, phase equilibrium, summation and energy balance (MESH) equations.

Physical and chemical properties for all species are taken from Aspen Plus component database. The reaction kinetics measured and evaluated in the laboratory kinetic studies is incorporated into the model.[7] The modeling of the decanter is achieved by the Decanter model of Aspen plus. The equilibrium constant is taken from Chiang et al and our previous work. [9, 10] The feed flow rate of both reactant, viz acetic acid and iso amyl alcohol is varied from 0.0175 kmol/hr to 0.07 kmol/hr and a feasible design is obtained. The reflux ratio is increased in order to reduce the catalyst volume required. The reboiler duty is also increased. The set conversion of iso amyl acetate (0.99) in the bottom is achieved with a reflux ratio of 2. Reflux has a twofold purpose in RD. Increasing reflux rate enhances separations and recycles unreacted reactants to the reaction zone and, thereby, increases conversion. As a result of the increasing reflux ratio several effects occurs in reactive distillation column: (i) the concentrations of reactants in the distillate are reduced; (ii) the reaction zone temperatures are reduced. It was observed that non-convergence of the simulation with higher value of reflux ratio.

When the catalyst volume is reduced, higher temperatures are found in the section closer to the condenser. Even though the reflux ratio and the condenser duty are increased with respect to the ideal design, the condenser did not cool the vapor leaving the top of the packing to ambient temperature. This is due to the smaller reaction volume as compared to the ideal case. Changing the number of stages in either the reactive or non-reactive zones, did not result in an improved design when the total height of reaction and stripping zone were kept constant. An increase in the number of stages resulted in flat mole fraction profiles, which indicated that these regions are accomplishing little to no separation. [12, 13]

Another important consideration is to know the system behavior at the operating conditions: existence of azeotropes, VLE, properties and residue curve maps, distillation line diagrams, if available, and reliable thermodynamic and kinetic models. All these tools will give a description of the system and will help to predict its performance. [13] Details for the physical properties and the phase equilibrium data of the iso-amyl acetate system are taken from our previous work [7, 10]. For every system, the final objective is to find a reaction zone and feed location that accomplishes the purity specifications of the products and maximizes the rate by allowing a high concentration of reactants in the reaction zone. The definition of the feed and reaction zone location will depend on how well the system behavior is known, the volatilities of the reactants and products, the specifications of the distillate and bottoms, the characteristics of the reaction, and other considerations described in more detail in Subawalla and Fair (1999).[12]

General Procedure for the Design of RD

Unlike conventional distillation, detailed design of RD (minimum number of stages, minimum number of reactive stages, feed locations etc.) is less apparent. [9] Sneesby et al, (1997) presented a design procedure for the ETBE RD system. [11] Subawalla and Fair (1999) proposed a general design procedure for RD. [12]

Chiang et al, (2002) outlined the general design procedure for reactive distillation, which is given in the following steps [9]:

1. Fix the number of reactive trays (N_{Rxn}), and guess the numbers of trays in the stripping and rectification sections (N_S and N_R, respectively).
2. Determine the minimum number of trays in the stripping and rectification section ($N_{S, min}$) from the short cut design with a desired specification of iso amyl acetate (99%) and set $N_S = 2 N_{S,min}$.
3. Increase the number of trays in the rectification section (N_R) until the top product specification (99% water) is met.
4. Repeat steps 2 and 3 until N_S converges (usually N_S does not change much for different N_R values)
5. Compute the total annual cost (TAC)
6. Change the minimum of reactive trays (N_{Rxn}) so that the TAC is minimized.

Design Options- Reaction Zone

When iso-amyl alcohol and acetic acid is fed to the RD unit, it contacts the catalyst and reacts selectively to form iso amyl acetate and water. Since water is the low boiling component and the lightest product, it will concentrate in the vapor phase and eventually leave the column as the major component of the distillate. In contrast, iso-amyl acetate is the high boiling component and it will concentrate in the liquid phase, descending through the column to be removed as the bottom product from the reboiler.[7]

At the beginning of this work, two designs were considered for reactive distillation in the system under study.

(a) In the first design, a column with a total condenser is selected. Preliminary simulations are performed and convergence was feasible. However, the design is discarded since in the condenser, two immiscible phases are formed, an aqueous phase i.e. almost pure water and an organic phase containing water, amyl alcohol and amyl acetate. When a total condenser is used, the overhead product is condensed, with a portion returned to column as reflux. It is difficult to maintain constant level of aqueous phase. This affects the reflux rate to the column and also affects the top temperature. General rules of thumb recommend the

selection of a condenser pressure that allows ambient temperature water as the coolant.[7,14]

(b) In the second design, a RD column with a partial condenser is selected. In this design, the condenser with decanter (Phase separator) is used so as to allow condensation of the water from the vapor stream entering the condenser, and organic phase from decanter is recycled to the column as reflux. In this case the aim of the liquid-liquid separator is not product enrichment, but a withdrawal of the aqueous phase and a recycle of non-converted organic components to the column. [7] The reaction zone is positioned as the middle section of the column. The positioning of the reaction zone within the column depends not only on the relative volatilities of the reactants and products, but also on the type of reaction.

The feed, consisting iso-amyl alcohol and acetic acid is fed directly to the reaction zone in order to maximize its concentration in this region. Simulations are performed to examine the effect of feed location. For optimum conversion of iso amyl acetate the best feed location is identified as stage 10 for iso amyl alcohol (from top of the reactive zone) and stage 15 for acetic acid. (from bottom of the reactive zone) Column diameter depends on the pressure drop, liquid and vapor loading and percentage of flooding allowed. The reactive distillation column diameter is set to 50 mm. Columns with variable diameters are rejected because the maximum divergences considering all column sections are about 8%. [13] For the non-reactive zone, initial estimates for the theoretical stripping stages and reflux ratio are taken from the short cut method simulations.

The short cut method for conventional distillation is employed with the Fenske equation for estimation of the minimum number of stages, the Underwood equation for minimum reflux estimation, the Gilliland correlation for the number of stages at finite reflux and the Fenske correlation for feed tray location.[14,15] An option to increase conversion in reactive distillation processes is to increase the column pressure and, thereby o increase the temperature in the reactive section leading to an enhancement of the reaction rate. This is not possible using T-63, ion exchange resin as catalyst for this reaction because the increase in the column pressure would result in temperatures inside the reactive section of the column that are significantly above 408 K, which is the maximum operating temperature for the catalyst recommended by the manufacturer. [16] Therefore column and condenser pressure is set at 1 atm.

Process Development and Simulation

Simulations with the equilibrium based model with bulk reaction kinetics in liquid phase (Model #C) are performed. For the reactive zone, the number of reactive stages is first specified as one, and the number of reactive stages is increased up to a maximum of 10. The initial estimate for the catalyst volume is obtained from reactor design for RD assuming reactive section to be a packed bed reactor (PBR). This represents the minimum catalyst volume required. This volume was split and distributed on the reactive stages. Since the conversion achieved was small, the catalyst volume was increased and the conversion increased to 0.99.

Figure 1 shows a basic process flow diagram and optimum design and operating parameters obtained for iso-amyl acetate synthesis via RD. Table 1 summarizes the feasible design obtained for the iso-amyl acetate case under study.

Table 1. Design and Operating Parameters for the iso-amyl acetate Reactive Distillation System [7]

Description	Equilibrium Stage Model # C
Total Number of Stages	27
Total packed height (m)	2.75 m
Number of Stages (reactive)	6
Number of Stages (stripping)	12
Number of Stages (rectification)	9
Reactive zone, height (m)	1 m
Stripping zone, height (m)	1 m
Rectification zone, height (m)	0.75 m
Column diameter (m)	0.05
Reflux ratio	2
Feed location for iso-amyl alcohol (Top of reactive zone)	10
Feed location for acetic acid (bottom of reactive zone)	15
Reboiler duty (kw)	1.2
Mole fraction of iso-amyl acetate (bottom)	0.99
Mole fraction of Water (top)	0.9915

Simulation Results

In this section simulation results for equilibrium stage model# A, model# B and Model # C are presented. For equilibrium model # B, we have simulated reactive distillation column without decanter and for Model # C with decanter. All the simulations are performed using steady state simulator. With reliable thermodynamic data, realistic operating conditions and the rigorous Aspen plus equipment models, one can simulate actual plant behavior. Implementation of model equations and all simulations were carried out using the model RADFRAC from the steady-state simulator Aspen plus, which is based on a rigorous equilibrium-stage model for solving the MESH equations. In these models liquid phase non-idealities are taken into account by the NRTL-model and the gas phase is assumed to be ideal. Since esterification reaction is carried out at atmospheric pressure, the vapor phase nonideality considered is the dimerization of acetic acid as described by the Hayden-O'Conell second virial coefficient. [7, 9] The Aspen plus built-in association parameters are employed to compute fugacity coefficients
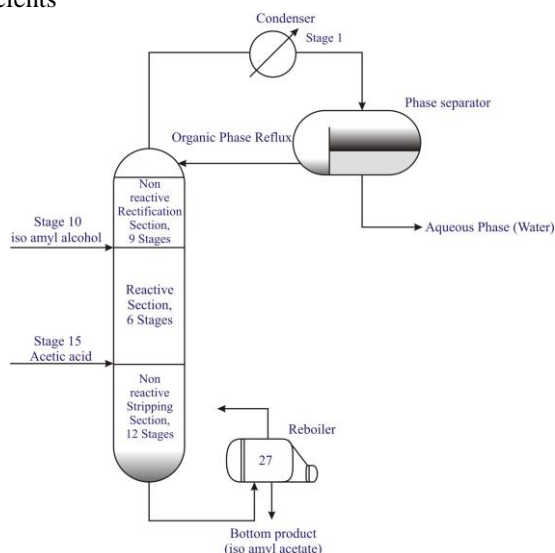

Figure 1. RD Process for synthesis of iso-amyl acetate [7,10]

Aspen plus Simulation:
Simulation Results for EQ stage Model # A:

The simulation was begun by putting together a basic flow sheet of a RADFRAC column with input streams of acetic acid and iso-amyl alcohol and output streams on iso amyl acetate and water. The basic process flow sheet for iso-amyl acetate synthesis in Aspen plus environment is as shown in Figure 2.
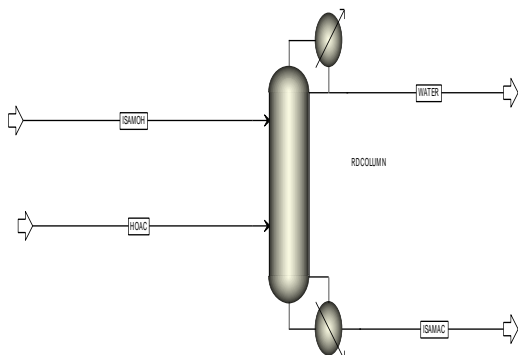
Figure 2. RADFRAC Model for iso-amyl acetate Synthesis (Model # A)

All the components that would be involved in the reactive distillation are chosen from the Aspen Plus component database. The NRTL model is selected as the property method for this simulation. The VLE data obtained from experiments is first put into separate data regression worksheets in the Aspen Plus user interface. Once this is done, Aspen Plus calculates the temperature dependent binary parameters for each of the six binary systems. Reaction rates for Pseudohomogenous model calculated by laboratory batch reactor are used for simulation to yield a steady state solution.

The feed location for iso-amyl alcohol and acetic acid is kept at stages 10 and 15 respectively with iso-amyl acetate being removed as bottom product at the 27th stage and the water as top product at the 1st stage.[7,10] The operating pressure of the column and the condenser pressure is also set at 1 atm.

**Base specification in Aspen plus Simulation [10]:**

Molar flow rates:

Feed 1 (Acetic acid)	:0.03200 kmol/hr
Feed 2 (iso amyl alcohol)	:0.03500 kmol/hr
Mole ratio (Acetic acid/ Alcohol)	:0.5384
Reboiler Duty	: 1.12 kW
Pressure	: 1 atm
Number of stages	: 27
Reactive zone stages	: 10-15
Stripping section stages	: 16-26
Rectification section stages	: 2-9
Feed stage location:	:
Feed 1 location (Acetic acid)	:15 (below reactive zone)
Feed 2 location (iso amyl alcohol)	:10 (above reactive zone)

It may be noted that all the base specifications given above is used for simulation of RD column for three different models, viz, Model # A, Model # B, Model # C in the following section. The composition profile obtained from Aspen plus simulation for four components, viz, acetic acid (HOAC), iso amyl alcohol (ISAMOH), iso-amyl acetate (ISAMAC) and Water (H_2O) are as shown in Figure 3.

Figure 3 shows the mole fractions of acetic acid, iso-amyl alcohol, iso-amyl acetate and water in the liquid phase. In the reaction zone (stages 10 to 15), iso amyl alcohol is consumed, and as a result, its mole fraction decreases.
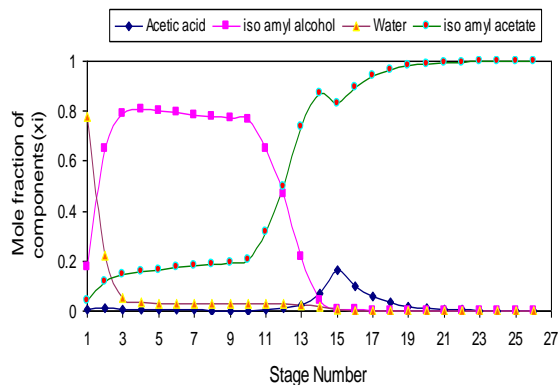
Figure 3. Steady State Composition Profile for Liquid Phase (Model #A)

Stages 15 and 16 show the transition between the reaction and stripping zones in the column. In the stripping zone, iso amyl acetate and iso amyl alcohol are being separated, resulting in the higher concentration of iso amyl acetate in the bottoms. High mole fractions of iso amyl acetate are obtained in the liquid phase. This is desirable since iso amyl acetate is the product of interest

Figure 4 shows the temperature profile obtained when the equilibrium model # A is employed. The reaction zone was located at the middle of the column and is comprised of stages 10 to 15. The remaining stages are pure separation stages. A smooth temperature profile is observed in the reactive zone where the catalyst is located. An important region is the reactive zone, where the catalyst is located.

In this region, the temperature profile is extremely important because temperature extremes in this region can accelerate deactivation of the catalyst and thereby, decrease its performance. It may be noted that the temperature range in the reactive zone is good enough for the thermal stability of catalyst. (135^0C). The larger temperatures in the bottom are due to the presence of iso-amyl acetate, which has a significantly higher molecular weight, and thus, a higher boiling temperature. (147^0C) [10]
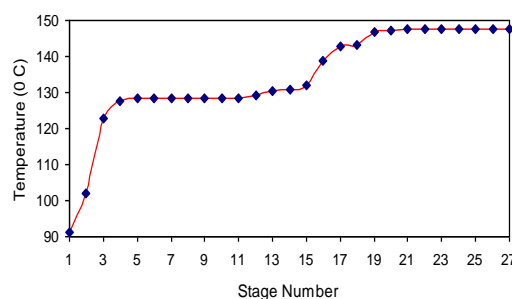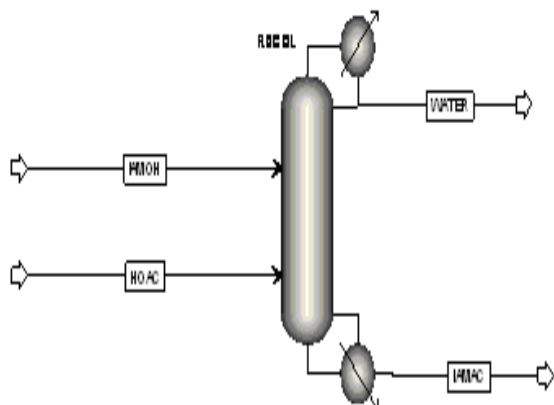
Figure 4. Steady State Temperatures Profile (Model # A)
Simulation Results for Model # B (Without Decanter)

In this model physical equilibrium is assumed on each stage and the reaction kinetics at each stage was described by a second order reaction. As discussed in Chapter 5, in the condenser, two immiscible phases are formed, an aqueous phase i.e. almost pure water and an organic phase containing water, amyl alcohol and amyl acetate. The organic phase is recycled back to column as reflux. However in this model does not account for decanter for separation of two immiscible phased that are formed in the condenser.

The simulation is begun by putting together a basic flow sheet of a RADFRAC column with input streams of acetic acid and iso-amyl alcohol and output streams are iso

amyl acetate and water. All the components that would be involved in the reactive distillation are chosen from the aspen plus component database. A Pseudohomogenous reaction kinetics model is used to describe the rate equation. The NRTL model is selected as the property method for this simulation. The basic process flow sheet for iso-amyl acetate synthesis in Aspen plus environment for this model is as shown in Figure 5. All the simulations are performed using steady state simulator.
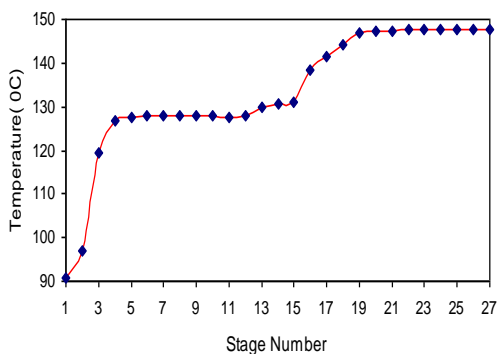

Figure 6. RADFRAC Model for iso-amyl acetate Synthesis (Model # B: Without Decanter)

The steady state composition profile for liquid phase is as shown in Figures 7. The steady state temperature profile is as shown in Figure 8.
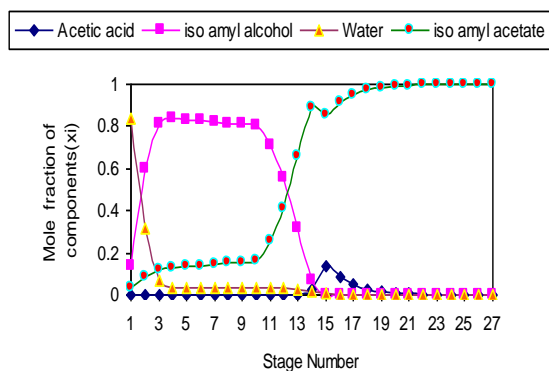
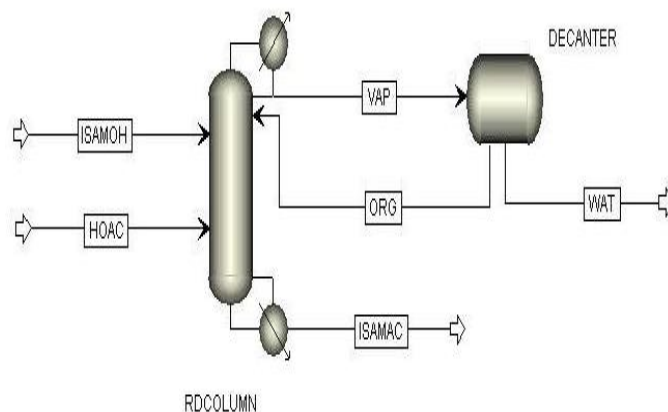**Figure 7. Steady State Composition Profile for Liquid Phase (Model # B: without Decanter)**

Figure 8. Steady State Temperature Profile (Model #B: without Decanter)

**Simulation Results for Model # C (With Decanter):**

In this model physical equilibrium is assumed on each stage and the reaction kinetics at each stage is described by a Pseudohomogenous reaction kinetic model. In this model, decanter is used for separation of two immiscible phased that are formed in the condenser. The basic process flow sheet for iso-

amyl acetate synthesis in Aspen plus environment for this model is as shown in Figure 9.


Figure 9. RADFRAC Model for iso-amyl acetate Synthesis (Model # C: With Decanter)

The iso-amyl acetate RD column is simulated using decanter. A Pseudohomogenous kinetics model is used to describe the rate equation. The decanter model of Aspen plus is used for the decanter simulation. All the simulations are performed using steady state simulator. The typical steady state composition profile for liquid phase is shown in Figure 10. The steady state temperature profile is as shown in Figure 11.
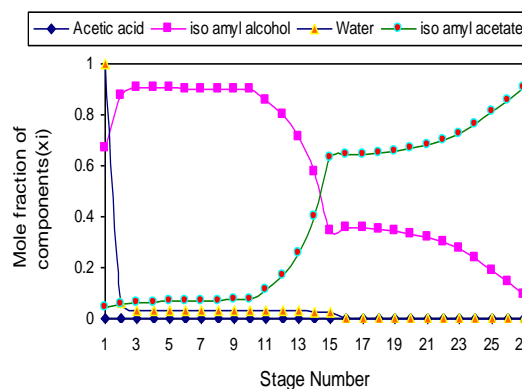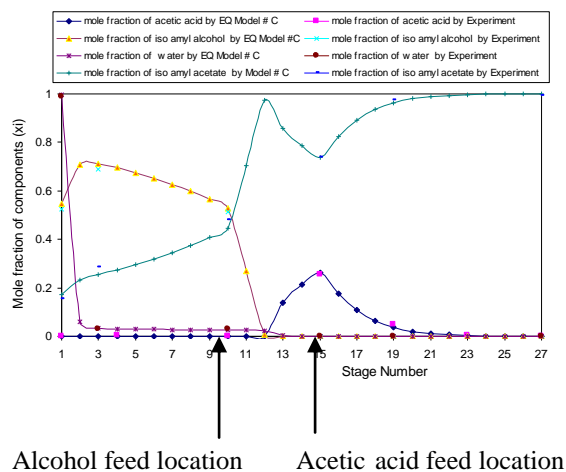

**Figure 10. Steady State Composition Profile for Liquid Phase (Model # C)**
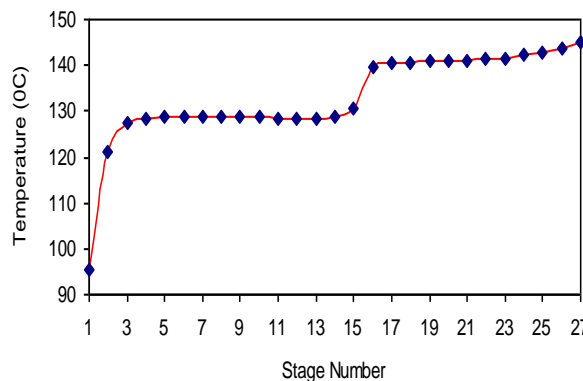
Figure 11. Steady State Temperature Profiles (Model # C) Comparisons of Experimental Results with Model Predictions (RUN NO # 10)

The model predictions were compared with experimental results for a typical representative RD run (Run No #10), which is as shown in Figures 12 and 13.

Figure 12 Comparisons of Aspen plus Simulation and Experimental Results for Composition Profiles (Run#10) (Model #C)
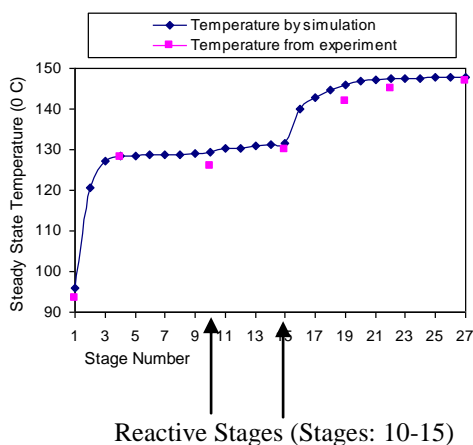
Figure 13 Comparisons of Aspen plus Simulation and Experimental Results for Temperature Profile (Run#10) (Model # C)

The temperature and composition profiles for this system from the equilibrium model are compared with experimental data under the same conditions. Comparison of the experimental data with simulation results indicated that an equilibrium stage model is capable of describing the column profiles quantitatively. The same model (Model # C with decanter) and simulator are used further to predict the performance of the column.

Model Validation

In order to use the RADFRAC model from Aspen plus for simulating a RD with confidence, the reliability of the model is tested by comparing the simulation results obtained with Model # C with the experimental data obtained on laboratory scale RD column. With the experimental results obtained under different operating conditions from our previous work are compared with results of the simulation run. It can be seen that the steady state composition profiles in the distillate and the bottom product and temperature profiles predicted by Aspen plus, are in good agreement with the experimental data.

The small difference may be due to the modified kinetic parameters that are based on the mole fraction rate expression Chiang et al, (2002) investigated the design alternatives for iso amyl acetate process with coupled reactor/ column and reactive distillation based on Total Annual Cost (TAC). [9] Teo et al, (2005) have studied the performance of the present system in RD column with dilute acetic acid. It is realized that the experimental

data on the system, pure acetic acid and iso amyl alcohol as feed to the column is limited and does not cover a wide range of operating parameters, such as reboiler duty, feed locations, feed flow rate, and molar ratio. Moreover, the column composition and temperature profiles, which helps to explain some important results, have not been presented in this paper.

Simulation Results for VLE user data and Aspen plus Data Bank:

The VLE data from the databank of Aspen plus is also used to simulate the RD column separately to observe how the results vary with the experimental data and the Aspen plus databank data. Figure 14 depicts the temperature profile of the the RD column.

Figure 14 shows the stagewise liquid composition profiles of the reactants and the products . As can be seen, the mol fraction of the iso-amyl acetate is 0.943 and that of water is 0.806
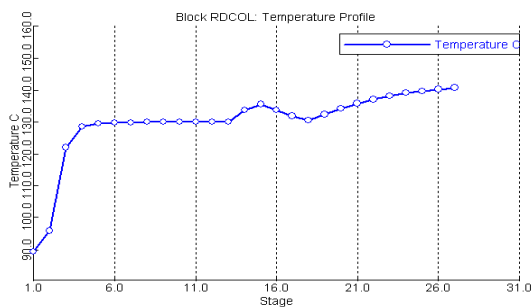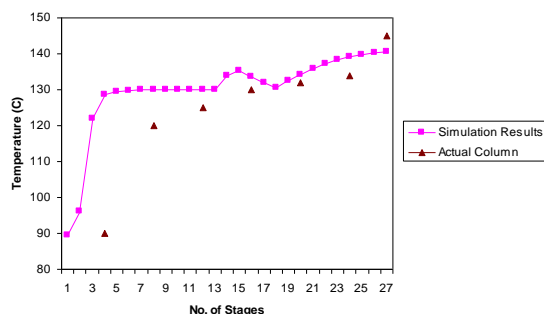

Figure 14 Stagewise Temperature Profile of the column
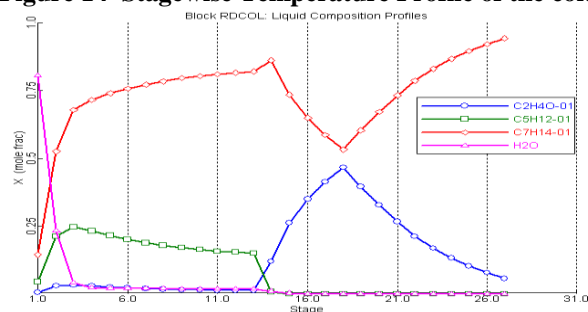
Figure 15 Stagewise Liquid Composition Profiles

As can be seen from the Table 3 and 4 and plots shown in figures 14 and 15, the simulation of the RD column has shown many conclusive results. Also as can be seen from Figure 16 , the temperature profile of the simulated column and the actual column increase at the same rate thus giving almost same results for the mol fraction of iso-amyl acetate in the bottoms and water in the overhead stream The mol fraction of iso-amyl acetate in the bottoms stream using databank data was 98% as compared to 94% using the user data. The experimental kinetic data also gave good results. There is a very good agreement between the results from the Aspen plus databank and the actual column.

Figure 16. Temperature profile of actual column and Simulation Results

Table 2 Details of the RD Experiment for Run No # 10 [7]

Sr. No.	Measurements	Value	Units
1	Feed Flow Rate (acetic acid)	0.03200	kmol/hr
2	Feed Flow Rate (iso amyl alcohol)	0.03500	kmol/hr
3	Mole ratio, acid/alcohol	0.5384	-
4	Reboiler duty	1.12	kW
5	Feed Location: (position on RD column)	iso amyl alcohol on position 5 (above reactive zone) acetic acid on position 7 (below reactive zone)	
6	Reactive Stages on RD column	Position 5 to 7	
7	Conversions in terms of acetic acid	99.50	%
8	Bottom product composition (mole fractions)	iso amyl acetate = 0.993	-
9	Top product compositions (mole fraction)	Water = 0.9845	-
10	Purity of bottom product	98.15	%

Table 3. Simulation Results using user data

RESULTS USING USER DATA Mole Fraction				
	Acetic acid stream	Bottoms stream	iso- amyl alcohol stream	Distillate stream
Acetic Acid	1	0.056437	0	0.004939
iso-amyl alcohol	0	6.27×10^{-7}	1	0.044621
iso-amyl acetate	0	0.943562	0	0.143504
Water	0	2.89×10^{-15}	0	0.806937

Table 4. Simulation Results using Aspen plus Databank data

RESULTS USING ASPEN PLUS DATABANK DATA Mole Fraction				
	Acetic Acid Stream	Bottoms stream	iso-amyl alcohol Stream	Distillate stream
Acetic Acid	1	0.01859889	0	0.03010285
iso-amyl alcohol	0	1.2265×10^{-8}	1	0.04281249
iso-amyl acetate	0	0.9814011	0	0.12821953
Water	0	4.3805×10^{-17}	0	0.79886512

Simulation Results for VLE User Data and Aspen plus Data Bank:

The VLE data from the databank of Aspen plus is also used to simulate the RD column separately to observe how the results vary with the experimental data and the Aspen plus databank data. The Table 5 shows the stagewise profile summary of the RD column. The figure 16 also depicts the temperature profile of the column.

Table 5. Final Result Comparisons

	Experimental Results	Simulation Results (Aspen Databank)	Simulation Results (User Data)
I D of column	50 mm	51 mm	51 mm
Mol.Fr. of iso-amyl acetate in Bottoms	0.983	0.981	0.943
Mol.Fr. of H_2O in Distillate	0.895	0.7989	0.807

Conclusion:

In this paper, design aspects of hybrid RD for the synthesis of iso-amyl acetate have been presented via steady state simulation for RD column. Processing strategies are identified through the extensive use of simulation tools employing three equilibrium-stage models of different complexity: Model #A, Model # B and Model # C. An accurate and efficient simulation of the reactive distillation process is successfully developed for the steady state case using the equilibrium (EQ) stage models # A, # B and # C. Model # C is used to demonstrate the design of iso-amyl acetate RD column. Predictions from stage models of different complexity are compared to the results of experiments. In Model #A, assumption of physical and chemical equilibrium shows that the additional assumption of chemical equilibrium on each stage is not valid. In Model # B (without decanter), taking the reaction kinetics into account yields better but still not satisfactory results. In Model # C, taking the reaction kinetics into account with decanter model, gives the simulation results of models agree with experimental results quantitatively. This indicates that the modeling depth of the simpler equilibrium-stage model with decanter (Model # C) is sufficient.

The model is validated with our own laboratory experimental data. The steady state simulation of RD column brings forth a number of important issues related to the optimal design techniques. In summary, RADFRAC model in Aspen plus is used for simulations of the iso-amyl acetate system using Model #C. A series of simulations are performed for the laboratory scale RD column, and a very good agreement between simulated and experimental data is obtained. Finally the VLE data from the databank of Aspen plus is also used to simulate the RD column separately to observe how the results vary with the experimental data and the Aspen plus databank data. It is observed that there is a very good agreement between the results from the Aspen plus databank and the actual column.

It should be noted that the external mass transfer resistance at the solid–liquid interface is not considered here. An exhaustive rate based model is required to be solved to consider these effects, which is out of the scope of the present work. Nevertheless the simplified model used here is able to explain the results over the conditions of interest.

Acknowledgements

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