Non–linear dynamic studies in Distillation columns: A Case study of industrial effluent

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Abstract. This work addresses the non-linear behaviour of distillation columns when there is an inappropriate selection of control variable in a system. An industrial effluent discharged from semi-conductor and pharmaceutical industries has been taken as a case study. A small disturbance in feed flow rate and feed composition has resulted in a ramp-like response of a recycle stream which has occurred due to the material imbalance in the system. This non-linearity in the process has been resolved by the selection of appropriate control variable.

1. Introduction
The first question that comes to our mind is why to control? What is this non-linearity in distillation columns? When we have a look at process industries, about 95% of the chemical industries are dealing with separation processes. The product purities, utility usage, and the process production rates are directly influenced by the distillation operations [1, 2]. Moreover, the overall economy of the plant is based on these factors. Control of distillation columns exhibiting non-linearity has become a thought-provoking problem for academicians. Non-linearity in distillation columns occurs because of the severe disturbances, non-stationary behaviour and multivariable coupling etc. [3, 4]. In fact, a non-linear system results in loss of convexity of the cost function to be minimised and a considerable increase in the calculations [5].

The effluents discharged from many chemical industries, especially pharmaceutical and Semiconductor industries consists of some close-boiling azeotropic mixtures which are very difficult to separate by conventional means of distillation. Tetrahydrofuran (THF)-water is one such effluent which has been taken in this case study. To separate this binary azeotropic mixture, some enhanced and cost-effective separation techniques need to be explored which can easily separate THF-water mixture. Extractive distillation is one such technique which can break the azeotropic point to separate the binary mixture [6, 7]. This technique uses the addition of a third component known as solvent/entrainer which alters the relative volatility of the base components of the binary mixture to break the azeotropic point. The recovery of entrainer in extractive distillation is the key step in making the process economical besides maintaining the product purities. The accumulation of entrainer either in the top or bottom streams of the distillation column leads to a decrease in the product purity levels. If the entrainer is not recovered fully, we can get impure products, rendering the process to be infeasible. These impurity levels in the overhead and bottom products vary asymptotically resulting in

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the static non-linearity in the distillation columns. Non-linearity in distillation columns occurs by a number of factors, especially when the columns are affected by a variety of disturbances, particularly in feed flow rates and feed compositions \([8, 9]\). A significant reduction in the variability of impurities in the products can lead to an improvised control of distillation columns. This can be achieved by coupling in which the composition of both overhead and bottom is to be controlled. This control performance affects the plant processing rates, product purities, and utility usage and hence determines the economic feasibility of the plant.

Notable works have been done by many researchers related to the material imbalance problems that occur in distillation columns or reactors. For example, Skogestad, Luyben and Ojasvi have worked on the selection of improper control structures which can lead to decrease in purity levels in distillation columns and low conversions in Reactors \([10, 11, 12]\). A critical review of distillation columns discussing the dynamic behaviour based on the non-linearity of high purity distillation columns resulting in the asymmetric dynamic response has been reported in the literature \([13]\). Few workers have worked on the non-linear dynamic behaviour of distillation columns due to the non-linear vapour-liquid equilibrium relationships \([14]\). Studies on transient behaviour of tray temperature resulting in non-linearity in a distillation column have been conducted by many researchers \([15]\).

Non-linearity in distillation columns also occurs if an appropriate control structure is not developed for the process. Any minor changes in the throughput can result in elevated flow rates of either product streams or recycle streams, affecting the product purities. In this case study, we have analysed the non-linear behaviour of a recycle stream in a distillation column separating a binary mixture of THF-water using N-hexane as an entrainer. A thorough dynamic analysis has been conducted in this case study which addresses the problem of non-linearity in the system when disturbances are induced in feed flow rates and feed composition which has resulted in a ramp-like response of the recycle stream because of inappropriate selection of the control variable. Later, an efficient control structure with appropriate control variable has been proposed which has settled the peculiar nature of recycle stream.

2. Simulation studies and Proposed Control structure

We have worked on the industrial effluent of Semi-conductor and Pharmaceutical industries which consists of a minimum-boiling azeotrope of THF-water. The mixture is separated by Extractive distillation technique using Hexane as an entrainer. The flow sheet has been developed in Aspen Plus and control studies have been carried out using Aspen Dynamics simulation software. The thermodynamic property package used is NRTL with binary interaction parameters obtained using Aspen Plus. The steady state flow sheet schematic is given in figure 1. From figure 1, we can see that an equimolar mixture of THF-water along with the two recycles (one as an organic phase coming from Decanter 1 and second as a distillate of Column 2) is mixed and is given as a feed at the stage no 12 to the Column 1. The two pure products, THF and water are obtained from the bottom of Column 1 and Column 2 respectively.
The minimum number of trays in both distillation columns is calculated using DSTWU analysis. Then the actual number of trays in the distillation column is taken thrice of the minimum number of trays as obtained from DSTWU analysis [16].

In order to develop an efficient control structure, we will first have a look on the column temperature profiles so that the temperature controllers are installed at appropriate tray locations. It is the temperature controller which plays a vital role in determining the robustness of a control structure for a process. If an appropriate temperature controller is not installed, we can get undesired product purities, disturbed flow rates etc. The temperature profile of two columns is given in figure 2.

From figure 2, we can observe that the most sensitive tray in column1 is 20th tray and that of column 2 is 3rd tray based on this sensitivity analysis for temperature control selection. We have proposed a control structure shown in figure 3. It consists of flow controllers, level controllers, pressure controllers and temperature controllers. The feed flow rate and feed composition are selected as a throughput manipulator and a disturbance of ±10% and ±5% is induced at 5hr in feed flow rate and feed composition respectively to check the robustness of the proposed control structure (Refer to figure 4). From figure 4, we can see that the recycle stream (R) is showing a ramp-like behaviour in all
the three components of THF, water and N-hexane and are not settling even if we carry the simulation run for more than 150hrs. Although, any disturbance in feed flow rate or feed composition induced in the steady state doesn’t reveal about this peculiar nature of recycle stream. Later, through the dynamic analysis, we have come to know about this non-linearity in the system. This work highlights the importance of dynamic studies which helps in determining the unusual response of recycle streams, which otherwise was not detected by Steady-state simulations.

![Proposed Control Structure](image)

**Figure 3.** Proposed Control structure.

![Ramp-like response of recycle stream](image)

**Figure 4.** Ramp like response of recycle stream for (a) THF, (b) water and (c) n-Hexane flow rates.

Looking at this ramp-like nature of all the components of recycle stream, we have to search for the reason why this non-linear behaviour of the system is there? Whether our proposed control structure is appropriate or not? To answer this question, we have carried out a thorough dynamic analysis by trying a variety of different control configurations to understand about this peculiar behaviour of recycle stream and have come to a conclusion that it is the incorrect selection of control variable which has resulted in this unusual nature of recycle stream. The tray temperature chosen was not appropriate and hence has resulted in non-linearity of the system i.e. for a ±10% or ±5% change in feed flow rate and feed composition, the recycle stream flow rate has shown a ramp-like behaviour. To encounter this problem of non-linearity, we have changed the position of tray temperature controller keeping rest of the control structure unchanged. Instead of controlling 20th and 3rd tray in
column 1 and column 2 respectively, we have selected tray no. 3rd in column 1 and 7th tray in column 2. On giving disturbance to feed flow rate and feed composition, the recycle stream has settled to its steady state value, showing that the non-linearity in distillation column has occurred due to the inappropriate control variable selection. The response of disturbances in feed flow rate and feed composition is shown in figure 5 and figure 6 respectively.

![Dynamic responses with ±10% disturbances in feed flow rate for improved control structure.](image)

**Figure 5.** Dynamic responses with ±10% disturbances in feed flow rate for improved control structure.
Figure 6. Dynamic responses with ±5% disturbances in feed composition for improved control structure.

3. Conclusions
This case study addresses the issue of non-linearity in the system which has occurred due to inappropriate selection of control variable. The selection of control variable becomes crucial to control the system when the distillation column is located within the recycle loop. Any disturbances in feed flow rate and feed composition results in undesired product purities due to the material imbalances within the system. So, one has to take care of the selection of correct control variable, otherwise, non-linearity in the system can occur resulting in the peculiar response of recycle streams and other product streams. The dynamic simulations have proved that it is just the temperature controller which has been installed at wrong tray location and has hence resulted in material imbalances within the process. Therefore, an appropriate control structure has been proposed which has improved the product purity, resulted in the reduction in energy consumption, better consistency, and increase in responsiveness.

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