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# SUSTAINABLE INTEGRATED PROCESS DESIGN AND CONTROL FOR AN EXTRACTIVE DISTILLATION COLUMN SYSTEM

Mohamad Zulkhairi Nordin, Mohd. Kamaruddin Abd. Hamid*

Process Systems Engineering Centre (PROSPECT), Faculty of Chemical Engineering,
Universiti Teknologi Malaysia
*Corresponding Author: kamaruddin@cheme.utm.my

## Abstract

The objective of this paper is to develop a sustainable integrated process design and control (*Sustain*-IPDC) methodology for an extractive distillation column system. Previously, IPDC methodology was developed and able to obtain an optimal solution for the IPDC problem for chemical processes in a simple and efficient way. However, the IPDC methodology did not consider sustainability aspect at the early chemical processes design stage. The methodology can be further extended to consider cost optimality, controllability and sustainability aspects. Here, the *Sustain*-IPDC problem for an extractive distillation column that is typically formulated as a mathematical programming (optimization with constraints) problem, is solved by decomposing it into six sequential hierarchical sub-problems: (i) pre-analysis, (ii) design analysis, (iii) controller design analysis, (iv) sustainability analysis, (v) detailed economics analysis, and (vi) final selection and verification. In the pre-analysis sub-problem, the concept of driving force is used to locate the optimal design-control-sustainability solution targets, which are defined at the maximum point of the driving force diagram. The performance of the methodology was analyzed for styrene – p-xylene separation process. The results show that the *Sustain*-IPDC methodology is capable of finding the optimal solution that satisfies design, control, sustainability and detailed economic criteria in systematic and efficient manner.

**Keywords:** Extractive distillation column; design; control; sustainability; optimization.

**Nomenclature**

| | | |
|---|---|---|
| $\alpha_{ij}$ | - | Relative separalibity parameter for component I respect tothe property j |
| **B** | - | Bottom flowrate |
| **d** | - | Set of disturbance variables |
| **D** | - | Distillate flowrate |
| **f** | - | Factor |
| **F** | - | Feed flowrate |
| $F_{Di}$ | - | Driving force |
| **G** | - | Category of objective function |
| **i** | - | Set of independent variables |
| **j** | - | Specific term of each category |
| **J** | - | Objective function |
| **L** | - | Liquid reflux flow |
| $M_i$ | - | Mass of substance i |
| $M_j^{(in)}$ | - | Inlet mass flowrate of stream j |
| $M_j^{(out)}$ | - | Outlet mass flowrate of stream j |
| $N_F$ | - | Feed Tray |
| **N** | - | Number of trays |
| **P** | - | Pressure |
| $P_{i,j}$ | - | Objective term |
| $P_1$ | - | Performance criteria for a separator design |
| $P_{2,1}$ | - | Sensitivity of x with respect to d |
| $P_{2,2}$ | - | Sensitivity of x with respect to u |
| $P_{3,1}$ | - | Sustainable objective mass consumption |
| $P_{3,2}$ | - | Sustainable objective energy consumption |
| $P_4$ | - | Economic objectives |
| **Qc** | - | Condenser duty |
| **Qr** | - | Reboiler duty |
| **RB** | - | Reflux boiler |
| **RR** | - | Reflux ratio |
| $RB_{min}$ | - | Minimum Reflux boiler |
| $RR_{min}$ | - | Minimum Reflux ratio |
| $T_B$ | - | Bottom Temperature |
| $T_D$ | - | Distillate Temperature |
| $T_F$ | - | Feed Temperature |
| **u** | - | Set of manipulated (design)variables |
| $u_d$ | - | Design variable |
| $u_m$ | - | Manipulated variable |
| **v** | - | Set of chemical system variables |
| **V** | - | Vapor flow rate |
| $w_{i,j}$ | - | Weight factor af each objective term |
| $w_{1,1}$ | - | Weight factor for design objective |
| $w_{2,1}$ | - | Weight factor for sensitivity objective |
| $w_{2,2}$ | - | Weight factor for controller structure objective |

| | | |
|---|---|---|
| $w_{3,1}$ | - | Weight factor for material consumption. |
| $w_{3,2}$ | - | Weight factor for energy consumption |
| $w_{4,1}$ | - | Weight factor for economic criteria. |
| $x$ | - | Set of process controlled variables |
| $x_{B,S}$ | - | Composition Styrene Bottom Column |
| $x_{B,X\text{-}DE}$ | - | Composition p-Xylene – Diethyl Ether Bottom Column |
| $x_{D,S}$ | - | Composition Benzene Top Column |
| $x_{D,X\text{-}DE}$ | - | Composition p-Xylene – Diethyl Ether Top Column |
| $x_i$ | - | Liquid phase composition of i |
| $x_{i,j}$ | - | Liquid mole fraction for component ion jth stage. |
| $X_{kj}$ | - | Mass fraction of component k |
| $x_S$ | - | Liquid composition of Styrene |
| $x_{X\text{-}DE}$ | - | Liquid composition of p-Xylene – Diethyl Ether |
| $Y$ | - | Set of binary decision variables |
| $y_S$ | - | Vapor composition of Styrene |
| $y_{X\text{-}DE}$ | - | Vapor composition of p-Xylene – Diethyl Ether |
| $y_i$ | - | Vapour phase composition of i |
| $y_{i,j}$ | - | Vapor mole fraction for component ion jth stage |
| $z_S$ | - | Feed composition of Styrene |
| $z_{X\text{-}DE}$ | - | Feed composition of p-Xylene – Diethyl Ether |
| $\theta$ | - | Set of constitutive variables |
| $\emptyset$ | - | Profit function |

## 1.0 INTRODUCTION

Chemical processes have been traditionally designed by a sequential approach consisting of initial process design, which is based on steady state economic calculations followed by the synthesis of a control structure that is generally based on heuristic controllability measures. Thus, the process design and process control aspects have been generally studied independently [1]. This traditional sequential design approach is often inadequate since the process design can significantly affect the process control [2-3]. Another drawback has to do with how process design decisions influence the controllability of the process. To ensure that design decisions give the optimum economic and the best control performance, controller design issues need to be considered simultaneously with the process design issues. The research area of combining process design and controller design considerations is referred here as integrated process design and controller design (IPDC).

Integrated Process Design and Control (IPDC) methodology was developed which is able to identify and obtain an optimal solution for the IPDC problem for chemical processes in an easy, simple and efficient way [4]. However, the developed methodology for the IPDC did not consider sustainability aspect in the early chemical processes design stage. Designing controllable and also sustainable process is one of the key challenges for sustainable development of chemical processes. Chemical process design can be further improved by including sustainability aspects within the developed IPDC method to ensure that the design is more cost efficient and controllable, as well as sustainable to meet product quality specifications. This can be achieved by extending the developed model-based IPDC method encompasses sustainability aspect.

Solving IPDC problem together with sustainability criteria may cause complexity in the optimization problem. Obtaining solutions for this problem will require a huge computational effort which makes this approach impractical for solving real industrial problems. In order to overcome the complexity of the IPDC problem and obtain an achievable optimal solution, a decomposition approach is used in this study. The decomposition approach has been applied in managing and solving the complexity of different optimization problems in chemical engineering [4]. The basic idea is that in optimization problems with constraints, the search space is defined by the constraints within which all feasible solutions lie and the objective function helps to identify one or more of the optimal solutions. In the decomposition-based approach, the optimization problem is decomposed into several sequential sub-problems [5]. The constraint equations are solved in a pre-determined sequence such that after every sequential sub-problem, the search space for feasible solutions is reduced and a sub-set of decision variables are fixed. When all the constraints are satisfied, it remains to calculate the objective function for all the identified feasible solutions to locate the optimal solution [6]. In this work, the decomposition solution strategy has been adopted to develop a new model-based methodology for solving IPDC problem.

Currently, a methodology known as sustainable integrated process design and control (*Sustain*-IPDC) for chemical processes had been developed [7]. This methodology is the improvised by including the sustainability analysis in the previous IPDC methodology.

Sustainability is frequently conceptualised as consisting of three factors, which are environment, society and economy. Essentially, sustainability is about the relationship between these three factors [8]. The relationship between these aspects shown graphically by a Venn diagram (Figure 1), where these factors are often symbolised as overlapping circles, and form three distinct of sustainability dimensions, which are one-dimensional (1D), two dimensional (2D) and three-dimensional (3D). 1D sustainability index means the sustainability index in terms of economic, environmental and social, are stand individually without interacting with each other. The individual index will be calculated and analysed individually. In addition, 2D sustainability index is calculated and analysed as based on the interactions between two sustainability criteria, which are socio-economic, socio-environmental and economic-environmental indices. On the other hand, the 3D sustainability index is obtained from the interaction between all the three sustainability criteria which is called the true sustainability.

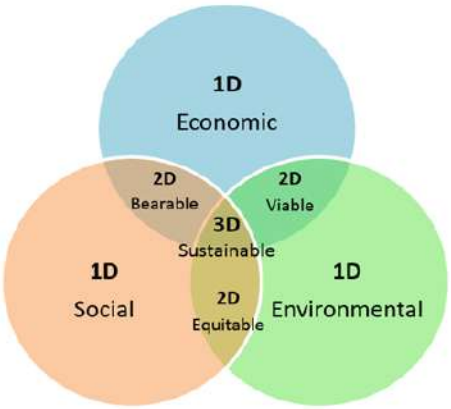


**Figure 1:** Venn diagram on the linkage among the sustainability criteria

One way to measure the sustainability is by calculating the sustainability index. In order to calculate the sustainability index, it is important to outline a set of indicators to characterize the extent of which the process is sustainable within each of the three different dimensions of sustainability. Indicators are used to assess the sustainability performance of a process or a system, in order to evaluate the progress toward enhancing the sustainability, and also to assist decision makers in evaluating the alternatives. Ideally, the chosen indicators should be

independent of each other, in addition to being small in number [9]. The independent requirement makes it easy to change the definition of some indicators or the way they are calculated, when suitable, according to the characteristics and type of data available, without affecting the others. Considering the three dimensions of sustainability in three distinct hierarchical groups [10]:

1) 1D indicators, which provide information about only one dimension of sustainability: economic, ecological, or societal [11].
2) 2D indicators, which provide information simultaneously about two dimensions of sustainability: socio-ecological, socio-economic, or economic-ecological [12].
3) 3D indicators or sustainability indicators, which provide information about all three dimensions of sustainability [13].

The objective of this paper is to describe in detail the methodology of sustainable IPDC (*Sustain*-IPDC) for an extractive distillation columns system, paying attention to encompass sustainability aspect to the current developed IPDC methodology. In the next section, the general formulation of a *Sustain*-IPDC is presented followed by the details description of a model-based methodology which is based on the decomposition approach for solving a *Sustain*-IPDC problem. After that, the important concepts used for obtaining the optimal sustainable design-control solution, are presented. Then, the application of the developed *Sustain*-IPDC in solving extractive distillation column design problems is presented followed by conclusion.

## 2.0 METHODOLOGY

### 2.1 Problem Formulation

The *Sustain*-IPDC problem is typically formulated as a generic optimization problem in which a performance objective in terms of design, controllability, sustainability and economic criteria is optimized subject to a set of constraints such as process (dynamic and/or steady state), constitutive (thermodynamics state) and conditional (process, control and sustainability specifications) constraints.

$$\textbf{max} \quad \boldsymbol{J} = \sum_{\boldsymbol{i=1}}^{\boldsymbol{m}} \sum_{\boldsymbol{j=1}}^{\boldsymbol{n}} \boldsymbol{P_{i,j} w_{i,j}} \tag{1}$$

Subject to:

Process (dynamic and/or steady state) constraint

$$\frac{dx}{dt} = f(\boldsymbol{u}, \boldsymbol{x}, \boldsymbol{d}, \boldsymbol{\theta}, Y, G) \tag{2}$$

Constitutive (thermodynamic) constraint

$$0 = g_1(\boldsymbol{v}, \boldsymbol{x}) - \boldsymbol{\theta} \tag{3}$$

Conditional (process, controllability, sustainability) constraint

$$0 = h_1(\boldsymbol{u}, \boldsymbol{x}) \tag{4}$$

$$0 \leq h_2(\boldsymbol{u}, \boldsymbol{x}, \boldsymbol{d}) \tag{5}$$

$$CS = \boldsymbol{x} + \boldsymbol{u}Y \tag{6}$$

The performance function of Eq. 1 includes the design, controllability, sustainability and economic. While Eq. 2 represents a generic process model from which the steady state model is obtained by setting dx/dt=0. Next, the constitutive equation that relates the constitutive variables (physical properties, reaction rate) is shown in Eq. 3 and the equality and inequality constraint are shown in Eq. 4 and Eq. 5. The equality and inequality constraint such as the product purity and others must be satisfied for a feasible operation, it can be linear or non-

linear. For Eq. 6, *Y* is for the controller structure selection in which corresponds whether a controller variable is paired with a particular manipulated variable or not.

The different optimization scenarios can be generated as follows:

- To achieve the process design objective which is the performance criteria for a distillation design, $P_1$ is maximized.
- To achieve the controller objectives, $P_{2,1}$ is minimized and $P_{2,2}$ is maximized. $P_{2,1}$ is the sensitivity of controlled variable, **y** with respect to disturbance, **d**. While, $P_{2,2}$ is the sensitivity of controlled variable, **y** with respect to manipulated variable, **u**.
- To achieve the sustainability objectives, $P_{3,j}$ is minimized. $P_{3,1}$, is the material consumption and $P_{3,2}$ is the energy consumption.
- To achieve the economic objectives, $P_4$ which is net profit function are need to be maximized.

The multi-objective function in Eq. (1) is then formulated as:

$$\max \; \boldsymbol{J = w_1 P_1 + w_{2,1}\left(\frac{1}{P_{2,1}}\right) + w_{2,2} P_{2,2} + w_{3,1}\left(\frac{1}{P_{3,1}}\right) + w_{3,2}\left(\frac{1}{P_{3,2}}\right) + w_4 P_4} \qquad (7)$$

**2.2 Decomposition-Based Solution Strategy**

The work flow and steps involved in the decomposition based solution strategy are shown in Figure 2. Accordingly, the *Sustain*-IPDC problem for an extractive distillation column is decomposed into six hierarchical stages: (1) pre-analysis; (2) design analysis; (3) controller design analysis; (4) sustainability analysis; (5) detailed economic analysis and (6) final selection and verification. As shown in Figure 2, the sets of constraint in the *Sustain*-IPDC problem for an extractive distillation column are decomposed into six sub-problems which correspond into six hierarchical stages. In this way, the solution of the decomposed set of sub-problem is equivalent to that of the original problem. As each sub-problem being solved, a large number of infeasible solution within the search space is identified and hence eliminated, thereby leading to a final sub-problem that is significantly smaller, which can be

solved more easily. Therefore, while the sub-problem complexity may increase with every subsequence stage, the number of feasible solutions is reduced at every stage.

***Stage 1: Pre Analysis***

At this stage, the objective is to define the operational window and set the target for the design-controller solution. Firstly, the **y** and **u** involved are analyzed and the important variables of **y** and **u** with the respect to the multi-objective function, Eq. (7) are shortlisted. The operational window is defined in terms of **y** and **u**. (note that **d** is known). Based on the thermodynamic and process insight and Eq. (3) (also defines the optimal solution targets), a choice is made for **y**. Then, the operational window is established by solving (for **u**) in Eqs. (4) and (5). For distillation design, the design target is selected based on the maximum points on the driving force (Figure 3). The maximum point on the driving force showed the maximum value of the desired product concentration. The plot of driving force usually has a well-defined maximum. It is important that from a process design point of view, the optimal objective (maximum value of $P_1$) can be obtained. For controller design point of view, at this target the controllability of the process is also best satisfied.

***Stage 2: Design Analysis***

The search space within the operational window identified in stage 1 is further reduced in this stage. The objective of this stage is to validate the target defined in the stage 1, by finding the acceptable value of **y** and **u** by considering Eq. (2) - steady state model. If the acceptable values cannot be found or the solution is located outside the operational window, then a new target is selected and the procedure is repeated until the suitable match is found.

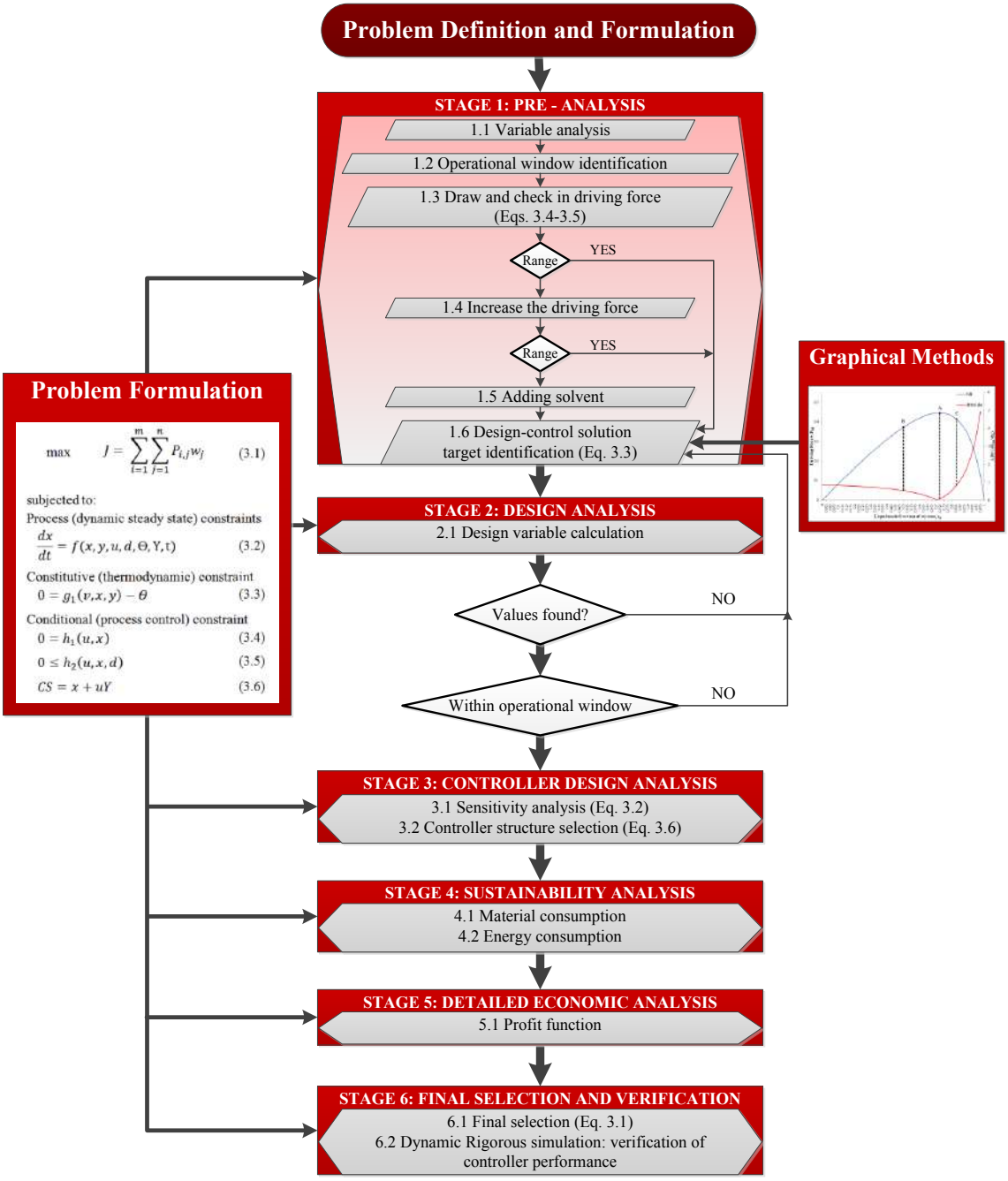


**Figure 2**: Sustainable Integrated Process Design and Control methodology for an extractive distillation system.

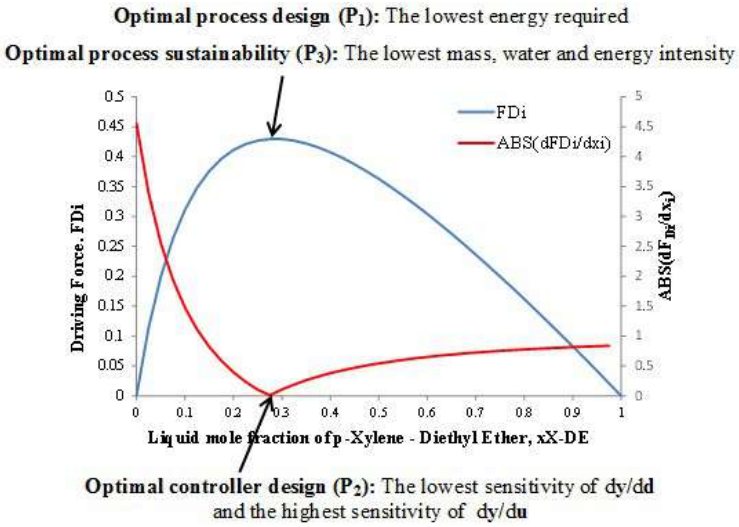


**Figure 3:** Determination of the optimal of design control for a separator using the driving force diagram at a specific pressure [14].

***Stage 3: Controller Design Analysis***

For this analysis, the search space is further reduced by considering the feasibility of the process control. This sub-problem considers the process model constraints, Eq. (2) (dynamic and/or steady state form) to evaluate the controllability performance of the controller candidates, and Eq. (6) for the selection of controller structure. For this controller design analysis there are two criteria that need to be analyzed which are: (a) sensitivity (***dy/dd***) of controlled variable **y** with respect to disturbances **d**, which in this case should be low, and (b) sensitivity *(**dy/du***) sensitivity of controlled variables **y** with the respect to the manipulated variables **u**, which should be high. Lower value of ***dy/dd*** means the process has lower sensitivity respect to the disturbances, makes the process more robust in maintaining its controlled variables against disturbances. Apart from that, best pair of the controlled and manipulated variables will be determined by the higher value of ***dy/du*** **(**to satisfy Eq. 6). Optimal design-process values become the set point for the controlled and manipulated variables according to the integrated design problem. So, the best set-point values of the controller need to be assumed by this methodology are actually those already defined as the design target (at maximum point of the driving force diagram).

***Stage 4: Sustainability Analysis***

For stage 4, the objective is to analyze the sustainability criteria which are based on three principle objectives, which are environmental protection, economic growth and social equity. There are metrics and indicator to be used to access the sustainability performance in the separator process in order to evaluate the progress toward enhancing sustainability, and to assist decision makers in evaluating alternatives. The sustainability analysis used in this work is based on the two-dimensional metrics, which are based on the simultaneously assessment of two out of the three sustainability dimensions. These include economic-environmental, socio-economic, and socio-environmental indicators [12].

***Stage 5: Detailed Economic Analysis***

The objective of this stage is to maximize the profit. The profit function, ϕ for the separator can be calculated as below [15]:

$$\emptyset = \boldsymbol{Product\ cost - Raw\ material\ cost - Operation\ cost} \\ \boldsymbol{-Depreciation\ cost} \quad (9)$$

***Stage 6: Final Selection and Verification***

At the final stage, the value of multi-objective function, Eq. (7) is analysed. The best candidate in terms of multi-objective function will be verified using rigorous simulation or experiment. It should be noted that rigorous simulation will be relatively easy because good estimations of **y** and **u** are obtained from stages 1 to 3. For stage 4, the value of sustainability is calculated by analysing the mass, water and energy intensity of the process. The detail economic analysis in stage 5 is analysed by finding its profit function, ∅. For controller performance, verification is made through open or closed loop simulations. For closed loop simulation, any tuning methods can be used to determine the value of control parameters.

**2.3 Defining Optimal Design Target**

The driving force (DF) concept used in this methodology in order to find the optimal design target to be use for the separation process and is given by the Eq. 10 [13]:

$$F_{Di} = y_i - x_i = \frac{x_i \alpha_{ij}}{1 + x_i(\alpha_{ij} - 1)} - x_i \quad (10)$$

From the driving force equation (Eq. 10), it shown that two-dimensional plots of $|F_{Di}|$ versus $x_i$ (or $y_i$) can be made as shown in Figure 4. The $BD_y$ and $AD_y$ lines represent operating lines corresponding to minimum reflux, while BD and AD represent operating lines intersecting on the line $D_y$ - $D_x$ for a reflux greater than the minimum. As $x_i$ approaching 0 or 1, the $|F_{Di}|$ approaching 0. The objective is to find the maximum condition for the operation of distillation column. However, there is case to obtain DF diagram with azeotrope or small DF value (Figure 5). This situation shows that it is hard to perform the separation process. Here, the extractive distillation column can be used where the addition of solvent is required. Adding the suitable solvent in the mixture would help remove/break the azeotrope or increase the DF value, the separation easier to perform.

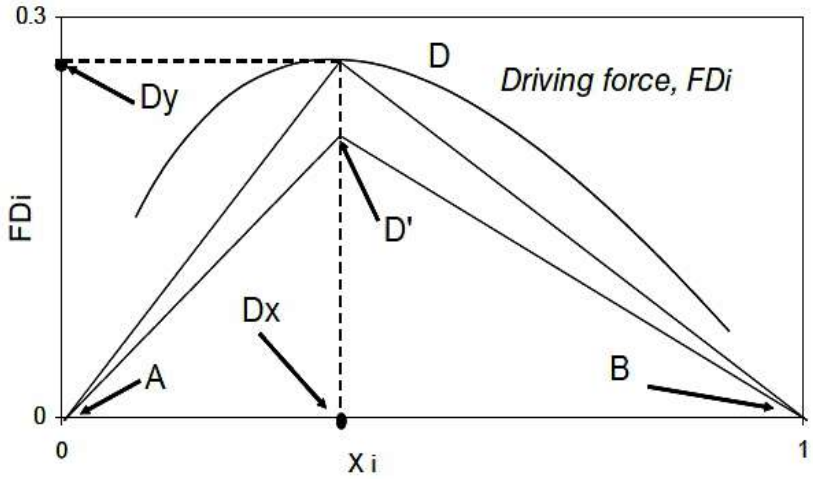


**Figure 4:** Driving force diagram for constant $\alpha_{ij} = 3$.

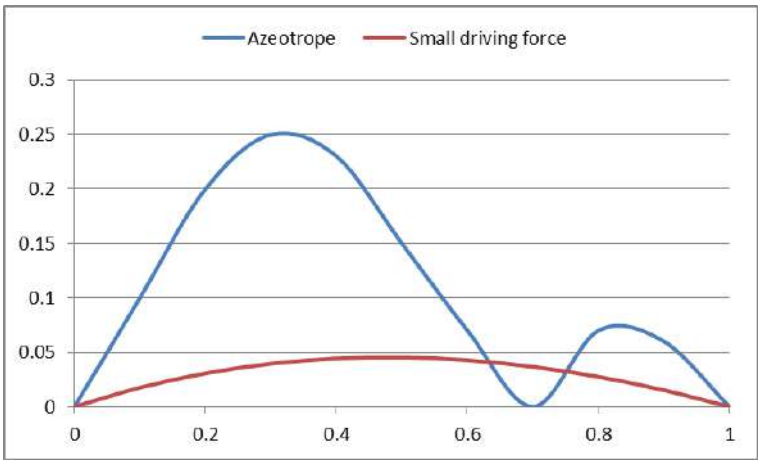


**Figure 5:** Driving force diagram for azeotrope and small driving force

The DF concept has an important role in solving the *Sustain*-IPDC problem. This concept is relatively straight forward to apply for finding the optimal solutions from process design, process control and sustainability. In Stage 1 of this methodology, targets for the design-control-sustainable solution are defined at the highest point of the DF diagram. Defining the target at the maximum point of the DF diagram ensure the optimal solution not only for the process design and control but also for the sustainable design.

## 3.0 *SUSTAIN*-IPDC METHODOLOGY FOR AN EXTRACTIVE DISTILLATION COLUMN CASE STUDY

### 3.1 Process Description

The application of the *Sustain*-IPDC is illustrated for the separation system of a styrene (S) – p-xylene (X) process. Consider a mixture of styrene and p-xylene. Both of these components have about similar molecular formula, molecular weight and boiling point but different chemical structures. The similar properties and the close relative volatility of both components make the separation becomes difficult and energy intensive. Therefore, it is important to consider the operation improvement in the design phase to increase the ease of separation of this mixture. One of the options is by using solvent to extract one of the components. A solvent is needed to increase the ease of separation of the mixture of styrene and p-xylene. Both components together with the solvent are fed into an extractive distillation

column where it is split into two streams of specified purity – distillate product (stream D with mainly p-xylene and the solvent) and bottom product (stream B containing 99% of styrene and 1% of the p-xylene and its solvent). A schematic of the process is depicted in Figure 6. All the solvent are assumed to be recovered after the process with a recovery column and then obtain the required product quality. The process is operated at a nominal operating condition as specified in Table 1. The objective is then to determine the sustainable design-control solution in which the multi-objective function with respect to design, control, sustainability and economic is optimal. This can be achieved by formulating the above problem as a *Sustain*-IPDC problem as below.

**Table 1**: Nominal operating conditions of the styrene – p-xylene separation process.

| Variable | Value | Description |
|---|---|---|
| $F$ | 10 | Feed flowrate (kmol/h) |
| $B$ | 3.469 | Bottom flowrate (kmol/h) |
| $T_F$ | 90 | Feed temperature (°C) |
| $P$ | 1 | Feed pressure (1 atm) |
| $z_{X\text{-}DE}$ | 0.65 | p-Xylene – Diethyl Ether feed molar composition |
| $z_S$ | 0.35 | Styrene feed molar composition |
| $N$ | 10 | Number of stages |

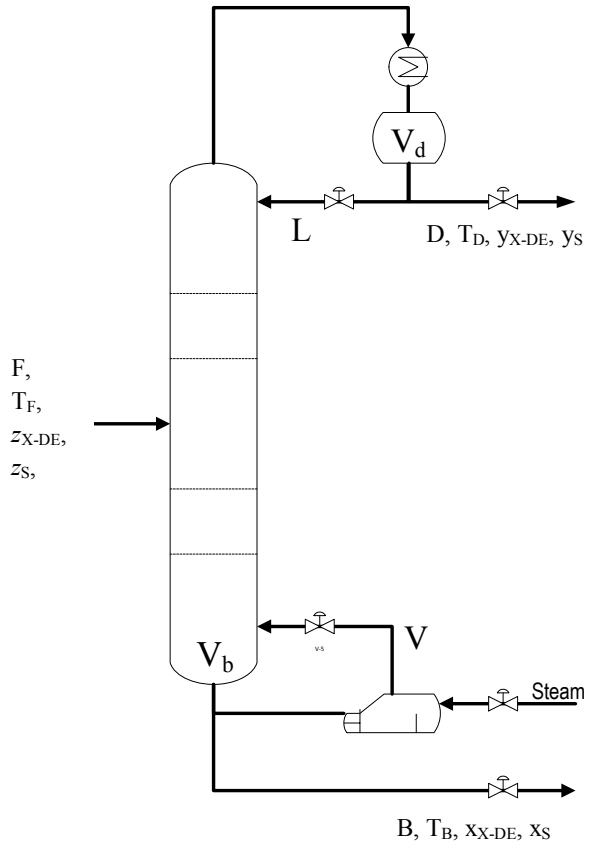


**Figure 6**: Extractive distillation column for Styrene – p-Xylene separation process.

### 3.2 Problem Formulation

The *Sustain*-IPDC problem for the process described above is defined in terms of a performance objective (with respect to design, control, sustainability and cost), and the three sets of constraints (process, constitutive and conditional).

$$\max \; \boldsymbol{J = w_1 P_1 + w_{2,1}\left(\frac{1}{P_{2,1}}\right) + w_{2,2} P_{2,2} + w_{3,1}\left(\frac{1}{P_{3,1}}\right) + w_{3,1}\left(\frac{1}{P_{3,1}}\right) + w_4 P_4} \tag{14}$$

subject to:

Process (dynamic and/or steady state) constraints

Total mass balance for each stage:

$$\frac{dM_1}{dt} = L_2 - V_1 - L_1 + F_1 \tag{15}$$

$$\frac{dM_j}{dt} = V_{j-1} + L_{J+1} - V_j - L_j + F_j \tag{16}$$

$$\frac{dM_N}{dt} = V_{N-1} - V_N - D - L_N + F_N \tag{17}$$

Component balance for each stage:

$$\frac{dM_{i,1}}{dt} = L_2 x_{i,2} - V_1 y_{i,1} - L_1 x_{i,1} + F_1 z_{i,1} \tag{18}$$

$$\frac{dM_{i,j}}{dt} = V_{j-1} y_{i,j-1} + L_{j+1} x_{i,j+1} - V_j y_{i,j} - L_j x_{i,j} + F_j z_{i,j} \tag{19}$$

$$\frac{dM_{i,N}}{dt} = V_{N-1} y_{i,N-1} - V_N y_{i,N} - D x_{i,N} - L_N x_{i,N} + F_N z_{i,N} \tag{20}$$

Energy balance for each stage:

$$\frac{dU_1}{dt} = L_2 h_2^l - V_1 h_1^v - L_1 h_1^l + F_1 h_1^f + Q_r \tag{21}$$

$$\frac{dU_j}{dt} = V_{j-1} h_{j-1}^v + L_{j+1} h_{j+1}^l - V_j h_j^v - L_j h_j^l + F_j h_j^f \tag{22}$$

$$\frac{dU_N}{dt} = V_{N-1} h_{N-1}^v - V_N h_N^v - D h_N^l - L_N h_N^l + F_N h_N^f - Q_c \tag{23}$$

Constitutive (thermodynamic) constraints

$$F_{Di} = y_{i,j} - x_{i,j} \tag{24}$$

$$y_{i,j} = \frac{\alpha_{i,jk} x_{i,j}}{1 + x_{i,j}(\alpha_{i,jk} - 1)} \tag{25}$$

$$\alpha_{i,jk} = \frac{K_{i,j}}{K_{j,k}} \tag{26}$$

$$K_{i,j} = K_i(T_j, P_j) \tag{27}$$

Conditional (process-control) constraints

$$x_{\mathrm{X}} \leq 0.01 \tag{28}$$

$$CS = \mathbf{y} + \mathbf{u}\mathrm{Y} \tag{29}$$

### 3.3 Decomposition-based Solution Strategy

The *Sustain*-IPDC problem for a distillation column system is decomposed into six hierarchical stages: (1) pre-analysis; (2) design analysis; (3) controller design analysis; (4) sustainability analysis; (5) detailed economic analysis and (6) final selection and verification.

#### *Stage 1: Pre-analysis*

The main objective of this stage is to define the operational window within which the optimal solution is located and set the targets for the optimal design-controller solution.

*Step 1.1: Variables analysis*

The first step in Stage 1 is to perform variable analysis. All variables involved in this process are analyzed and classified as design and manipulated variables **u**, process controlled variables **y**, and disturbances **d** as shown in Table 2. Then, the important **u** and **y** are selected with respect to the multi-objective function, Eq. (14), and tabulated in Table 3.

**Table 2**: List of all design and manipulated variables, process-controlled variables and disturbances for a styrene – p-xylene extractive distillation column.

| | |
|---|---|
| Design variable ($\mathbf{u}_d$) | $N_F$, $RR$, $RB$, $Q_r$, $Q_c$ |
| Manipulated variable ($\mathbf{u}_m$) | $B$, $D$, $V$, $L$ |
| Process-Controlled variables ($\mathbf{y}_m$) | $x_{D,X\text{-}DE}$, $x_{D,S}$, $x_{B,X\text{-}DE}$, $x_{B,S,}$ $T_D$, $T_B$ |
| Disturbances (**d**) | $T$, $z_{X\text{-}DE}$, $z_S$, |

**Table 3**: List of important design and manipulated and process-controlled variables for a styrene – p-xylene extractive distillation column design.

| | |
|---|---|
| Design variable ($\mathbf{u}_d$) | $N_F$, $Q_r$, $Q_c$ |
| Manipulated variable ($\mathbf{u}_m$) | $V$, $L$ |
| Process-Controlled variables ($\mathbf{y}_m$) | $x_{D,S}$, $x_{B,S}$ |

*Step 1.2: Operational window identification*

The operational window is identified based on bottom and top product purity. Since it is desired to have pure bottom product, the quality at bottom product should be monitored and controlled. On the other hand, most of the top product consists of p-xylene and the diethyl solvent. Their purity will not be monitored and controlled because this stream need to undergo another process to separate between the p-xylene and the diethyl solvent to recovered the p-xylene. In order to satisfy the product quality, the bottom p-xylene – diethyl ether composition should be less than 0.01.

*Step 1.3: Draw and check the driving force*

The step-by-step algorithm for a simple distillation column proposed by Gani and Bek-Pederson is implemented here [13]. The driving force diagram for the styrene – p-xylene system at P = 1 atm is drawn as shown in Figure 7. Driving force is a measure of the relative ease of separation. The larger the driving force, the easier the separation is. From Figure 7, the value of driving force for the system is less than 0.1, which means that the styrene – p-xylene system is categorized as a difficult separation and can implement the proposed methodology.

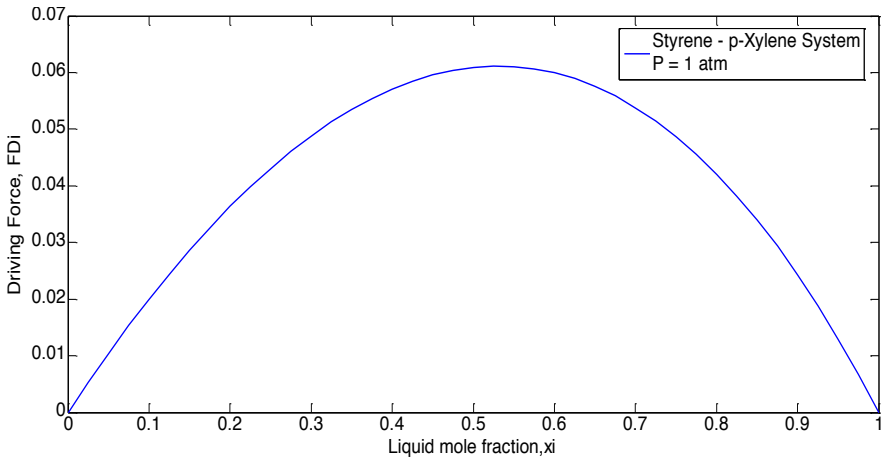


**Figure 7:** Driving force diagram for separation of Styrene – p-Xylene by an extractive distillation column at P = 1 atm.

*Step 1.4: Increase the driving force*

Based on Eq 24. – Eq. 27, the driving force can be increased by changing the operating conditions either temperature and/or pressure. Changes of temperature must be within its allowable range of temperature. For the styrene – p-xylene system, as shown in the Figure 8, there is no significant change of the driving force is observed. In addition, the driving force value is still less than 0.1, meaning it is still difficult to separate the mixture. Hence, we need to proceed to the next step.

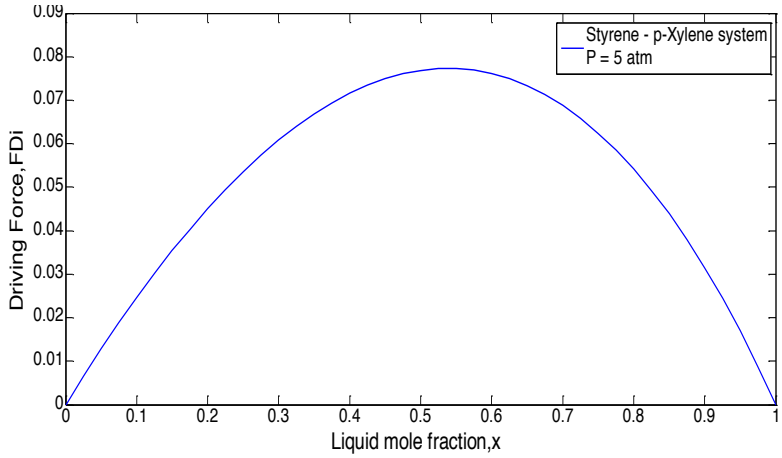


**Figure 8**: Driving force diagram for separation of Styrene – p-Xylene by an extractive distillation column at P = 5 atm.

*Step 1.5: Adding solvent*

Solvent is added with the purpose of absorbing and mix but does not react with one of the component. When the solvent absorbed and combines with the component, they will become a new component with a new property. In this study, the property of the new component is defined as the average of the component and solvent properties. There are five solvents that have been selected which are diethyl ether, chloroform, hexane, benzene and toluene. Figure 9 shows the driving force diagram before and after adding the solvent.

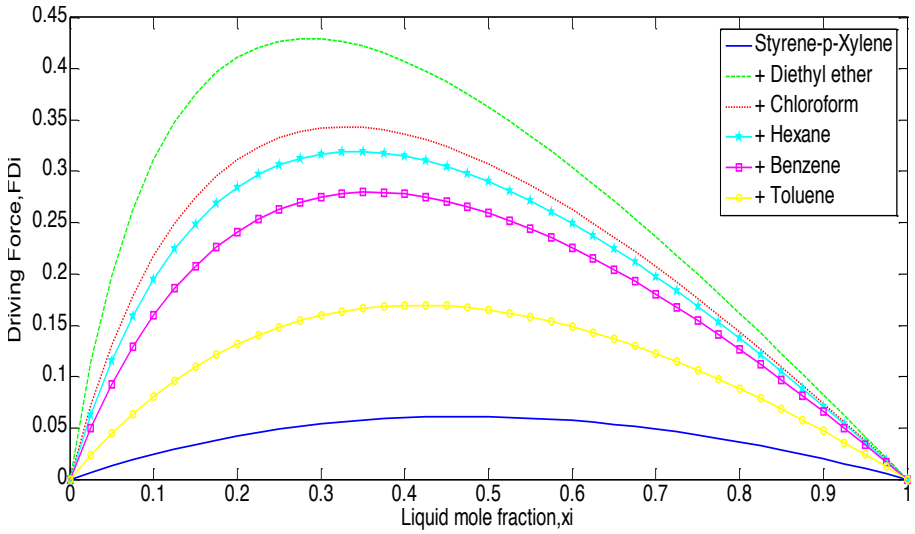


**Figure 9**: Driving force diagram for separation of Styrene – p-Xylene with different solvents by an extractive distillation column.

It can be seen in Figure 9 that, adding solvents will drastically increase the driving force to more than 0.1, which means that normal separation cannot be performed. Furthermore, Figure 9 shows that different solvent has different effect in terms of the increment of the driving force value. Since adding diethyl ether solvent will increase the driving force the most, therefore, diethyl ether is chosen as the solvent for the styrene – p-xylene separation process. The diethyl ether solvent is chosen because it gives the highest driving force for the styrene – p-xylene system.

*Step 1.6: Design-control target identification*

The driving force diagram for the Styrene - p-Xylene with diethyl ether solvent system at P = 1 atm is drawn as shown in Figure 10. In this graphical method, the target for the optimal process-controller design solution for an extractive distillation column is identified at the maximum point of the driving force (Point A) (see Figure 10). In Figure 10 also, two other points which are not at the maximum are identified as alternative candidates. From a process design point of view, they are not optimal since at this points the value of the driving force is smaller hence separation at this point is more difficult. Therefore, from a design perspective, Point A is an optimal solution for extractive distillation column design (this claim will be verified at Stage 4).

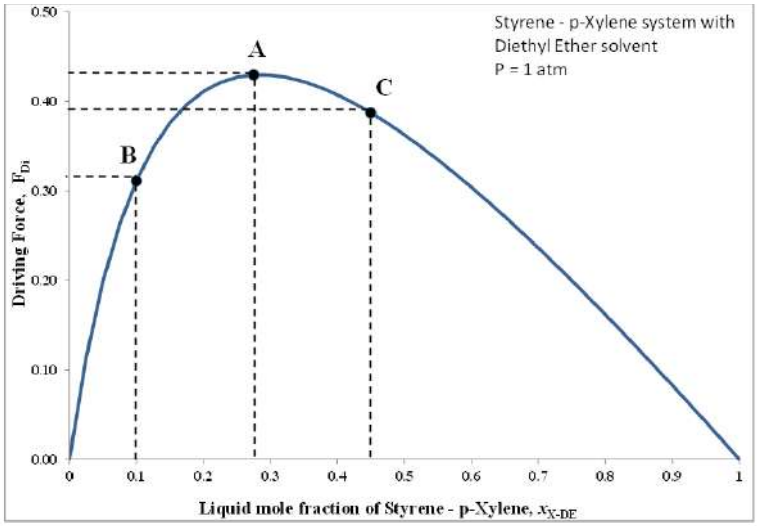


**Figure 10:** Driving force diagram for separation of Styrene – p-Xylene with Diethyl Ether solvent by an extractive distillation column.

***Stage 2: Design analysis***

The objective of this stage is to validate the target identified in Stage 1 by finding the acceptable values of **y** and **u**. In this stage, the search space defined in Stage 1 is further reduced.

*Step 2.1: Design-manipulated and process-controlled variables value calculation*

The established targets (points A, B, C) in Figure 10 are now matched by finding the acceptable values of design variables (e.g. feed stage, $N_F$ and reflux ratio, $RR$). The values of the design variables are determined graphically as shown in Figure 11. Table 4 summarizes the results obtained graphically with respect to design variables for three different design alternatives. With specified values of $N$, $N_F$, $RR$, product purity and feed conditions, the design of distillation column is verified using the Aspen HYSYS process simulator, in order to find values of other design-process variables. Results of the steady state simulation for different design alternatives are tabulated in Table 5. It is noted that design at the maximum point of driving force (Point A) corresponds to the minimum with respect to energy consumption compared to other points. The design at the maximum point of driving force (Point A) should correspond to the minimum of energy consumption compared to other points [16]. However for this case, the Point C has the minimum energy consumption followed by the Point A and Point B which consumed the largest amount of energy. Nevertheless, this does not indicate that Point B is the best design.

**Table 4:** Values of design variables for different design alternatives of styrene – p-xylene extractive distillation column design.

| Point | Design Variables | | | | | |
|---|---|---|---|---|---|---|
| | $N$ | $N_F$ | $RR_{min}$ | $RB_{min}$ | $RR$ | $RB$ |
| A | 10 | 7 | 0.62 | 1.42 | 0.75 | 1.70 |
| B | 10 | 9 | 0.45 | 3.08 | 0.54 | 3.70 |
| C | 10 | 6 | 0.71 | 0.65 | 0.86 | 0.78 |

***Stage 3: Controller design analysis***

The objective of this stage is to evaluate and validate that the controllability performance of the feasible candidates in terms of their sensitivities with respect to disturbances and manipulated variables.

**Table 5**: Steady state simulation results for different design alternatives of styrene – p-xylene extractive distillation column design.

| Variable | Point A | Point B | Point C |
|---|---|---|---|
| **Feed** | | | |
| $F$ (kmol/h) | 10 | 10 | 10 |
| $T$ (°C) | 90 | 90 | 90 |
| $P$ (atm) | 1 | 1 | 1 |
| $N_F$ | 3 | 5 | 2 |
| $z_{X\text{-}DE}$ | 0.65 | 0.65 | 0.65 |
| $z_S$ | 0.35 | 0.35 | 0.35 |
| **Distillate** | | | |
| $D$ (kmol/h) | 6.531 | 6.531 | 6.531 |
| $L$ (kmol/h) | 5.281 | 13.170 | 3.251 |
| $T_D$ (°C) | 86.56 | 86.56 | 86.56 |
| $x_{X\text{-}DE}$ | 0.99 | 0.99 | 0.99 |
| $x_S$ | 0.01 | 0.01 | 0.01 |
| $Q_c$ (kJ/h) | 362,000 | 604,000 | 300,000 |
| **Bottom** | | | |
| $B$ (kmol/h) | 3.469 | 3.469 | 3.469 |
| $V$ (kmol/h) | 10.610 | 17.100 | 8.395 |
| $T_B$ (°C) | 144.4 | 144.4 | 144.4 |
| $x_{X\text{-}DE}$ | 0.01 | 0.01 | 0.01 |
| $x_S$ | 0.99 | 0.99 | 0.99 |
| $Q_r$ (kJ/h) | 395,000 | 637,000 | 333,000 |

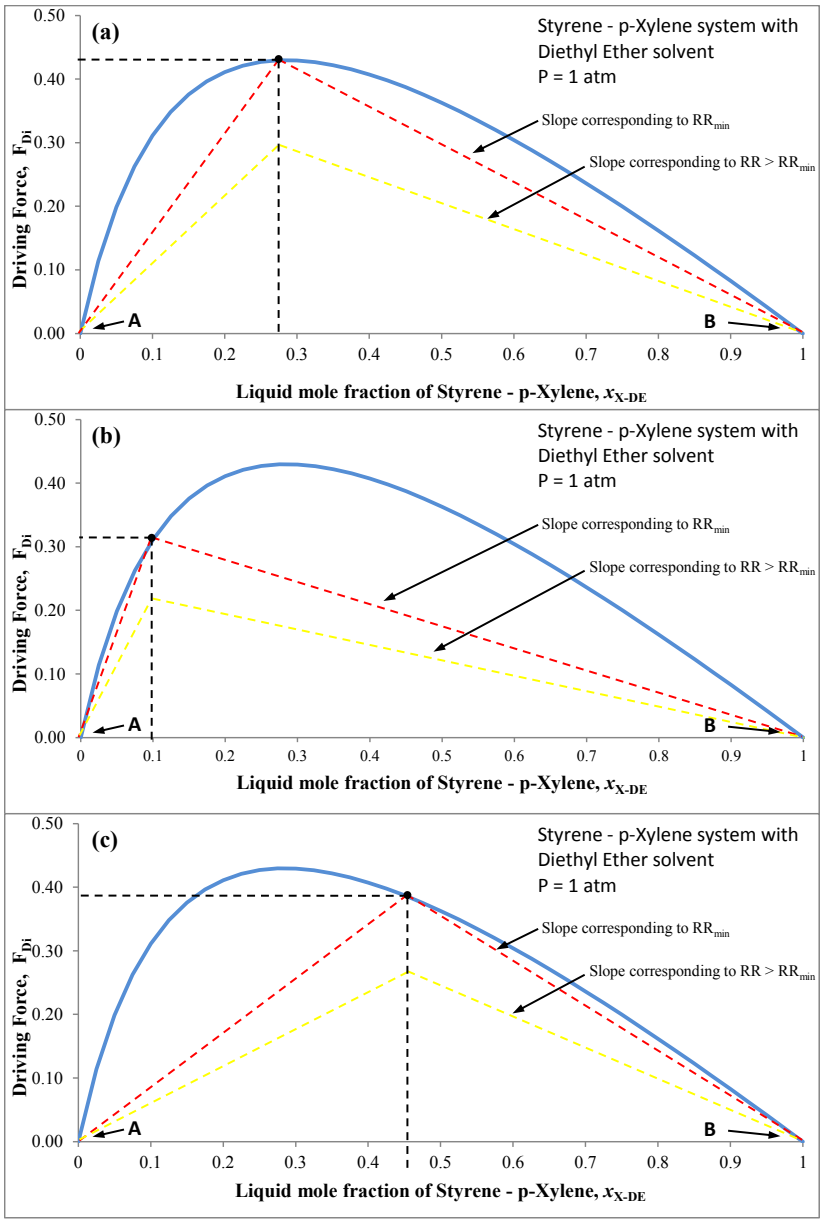


**Figure 11:** Driving force diagram with illustration of the distillation design parameters at: (a) Point A; (b) Point B; and (c) Point C for the separation of Styrene – p-Xylene with Diethyl Ether solvent.

*Step 3.1: Sensitivity analysis*

The process sensitivity is analyzed by calculating the derivative values of the controlled variables with respect to disturbances ***dy/dd*** with a constant step size using the process

simulator. The derivative of $F_{Di}$ with respect to $x_{X\text{-}DE}$ and values of derivatives for different designs are given in Figure 12 and Table 6.

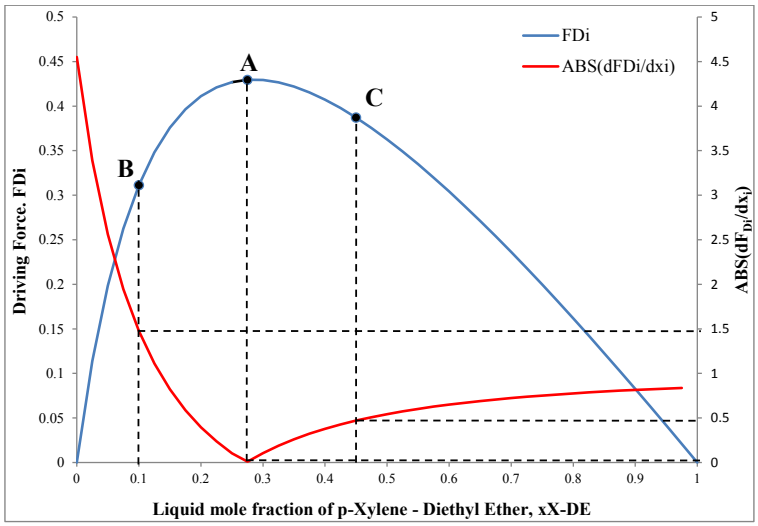


**Figure 12**: Driving force diagram for separation of Styrene – p-Xylene with Diethyl Ether solvent by an extractive distillation column and corresponding derivatives of the driving force with respect to composition.

**Table 6**: Derivatives values of $F_{Di}$ with respect to $x_{X\text{-}DE}$ at different distillation designs for styrene – p-xylene separation system.

| **Distillation Design** | **Derivative** $dF_{Di}/dx_{X\text{-}DE}$ |
|---|---|
| A | 0.1036 |
| B | 1.9446 |
| C | 0.4268 |

It can be seen that the derivative values are smaller for distillation design A compared to other designs (B and C). A smaller value of the derivative means that the process sensitivity is lower [17]. Hence, from a process control point of view, separator design A is less sensitive to the effect of disturbances which makes it more robust in maintaining its controlled variables in the presence of disturbances.

*Step 3.2: Controller structure selection*

According to Russel *et al*. (2002), $dx_{B,X\text{-}DE}/dV$ and $dx_{D,S}/dL$ can be represented as:

$$\frac{dx_{B,X-DE}}{dV} = \left(\frac{dx_{B,X-DE}}{dF_{Di}}\right)\left(\frac{dF_{Di}}{dx_{X-DE}}\right)\left(\frac{dx_{X-DE}}{dT_b}\right)\left(\frac{dT_b}{dV}\right) \approx \mathbf{0} \quad (4.17)$$

$$\frac{dx_{D,S}}{dL} = \left(\frac{dx_{D,S}}{dF_{Di}}\right)\left(\frac{dF_{Di}}{dx_S}\right)\left(\frac{dx_S}{dT_b}\right)\left(\frac{dT_b}{dV}\right) \approx \mathbf{0} \quad (4.18)$$

Since $dx_{B,X\text{-}DE}/dV \approx 0$ and $dx_{D,S}/dL \approx 0$, it is possible to maintain the $dx_{B,X\text{-}DE}$ and $dx_{D,S,}$ at their optimal set pint using either composition control and temperature control

Therefore, for this study a composition control will be implemented because it is a direct control approach which is easier to control. The controller structure is selected by calculating the derivatives values of the potential controlled variables ($x_{D,X\text{-}DE}$, $x_{D,S}$, $x_{B,X\text{-}DE,}$ $x_{B,S}$) with respect to manipulated variables (V and L). The controller structure is analyzed and calculated by changing the manipulated variables (V and L) in a constant step size for each distillation design point. The objective is to identify the best controller structure by pairing the controlled- manipulated variables which can satisfy the control objective (maintain the top and bottom purity which represent by the $x_{B,X}$-DE and $x_{D,S}$, purity at their optimal set point with the respect of disturbances).The values of derivatives for the different designs are tabulated in Table 7 and Figure 13.

**Table 7**: Derivative values of potential controlled variables with respect to potential of manipulated variables at different distillation designs for a styrene – p-xylene separation system.

| **Distillation** | **Derivatives with respect to V** | | **Derivatives with respect to L** | |
|---|---|---|---|---|
| **Design** | $dx_{D,S}/dV$ | $dx_{B,S}/dV$ | $dx_{D,S}/dL$ | $dx_{B,S}/dL$ |
| A | 0.0008 | 0.0860 | 0.0125 | 0.0352 |
| B | 0.0001 | 0.0788 | 0.0208 | 0.0153 |
| C | 0.0054 | 0.0853 | 0.0301 | 0.0289 |

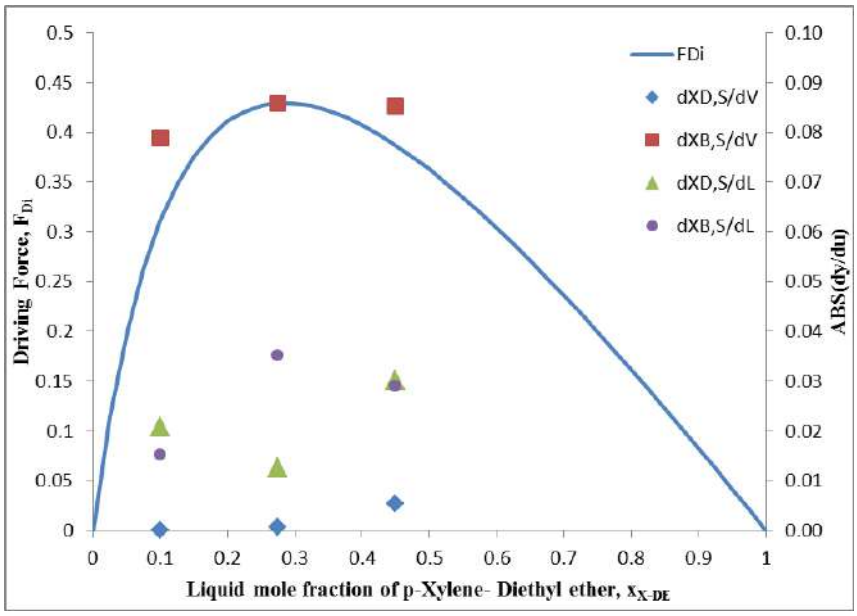


**Figure 13**: Driving force diagram for separation of Styrene – p-Xylene with Diethyl Ether solvent by an extractive distillation column with corresponding derivatives of $x_{D,S}$ and $x_{B,S}$ with respect to V and L.

From the Table 7 and Figure 13, it can be clearly seen that derivative of values of $dx_{D,S}/dL$ and $dx_{B,S}/dV$ are higher compare with other derivatives. As stated by Hamid (2010), the process has higher gain which in process control means it will require a smaller control action to maintain the controlled variable at the optimal set point value. Therefore it can be clearly seen from Table 7, that the best pairing of controlled-manipulated variable that will able to maintain product purity at the bottom of the distillation column is $x_{B,S}$-$V$, whereas the best pairing for controlling product purity at the top of the extractive distillation column is $x_{D,S}$-$L$. Figure 14 shows the proposed controller structures for controlling the top and bottom product purity. Both controller structures are direct feedback controllers. These controller structures will require less control action in maintaining column product purity. It should be noted that, at point A, the controller action and performance are at the best.

*PERINTIS E-Journal*, **2014**, Vol. 4, No. 2, pp.43-77

***Stage 4: Sustainability Analysis***

The objective of this stage is to identify the optimal design of distillation column in terms of sustainability. Several criteria will be measured in this stage to fulfill sustainability objective functions with respect to material and energy consumption.

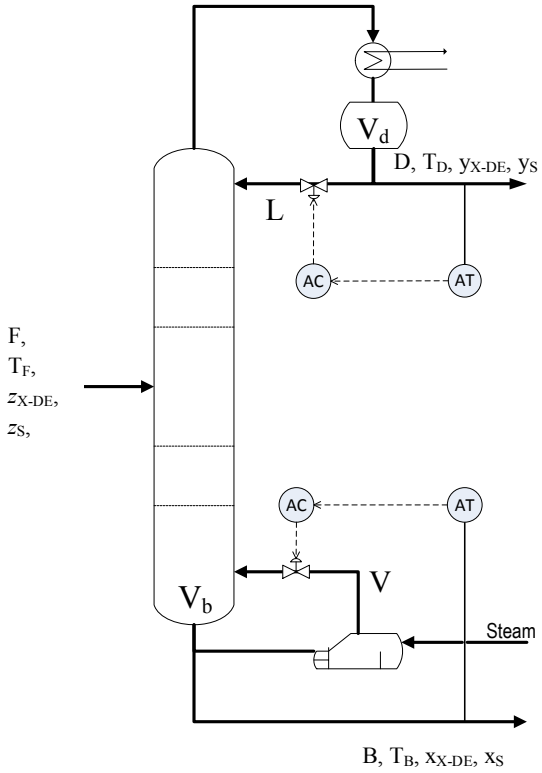


**Figure 14**: Controller structures for controlling the top and bottom product purity of the extractive distillation column for Styrene – p-Xylene separation process.

*Step 4.1: Material Consumption*

In this stage, the impacts of each design alternatives of distillation design to the sustainability is assessed by using two dimensional metrics [12]. The two dimensional metric those used in this stage are based on economic environmental indicators. In order for a distillation design to be sustainable, it must have lower impacts to the economic losses as well as environmental effect. Therefore, material consumption of each of the distillation column designs (Point A, Point B, Point C) is identified in order to find out the optimal design. By finding out the feed stream and the distillate in each point of distillation column design, the mass intensity index can be calculated. Furthermore, in distillation process, water is being used in the condenser to

cool down the distillate and as a steam to heat up the solution in the bottom. By assuming the amount of water usage in the distillation design, the water intensity index can be identified. The calculated values of sustainability metric are tabulated in Table 8.

**Table 8:** The values of mass and water intensity index at different distillation design for styrene – p-xylene separation system.

| Distillation Design | Mass Intensity Index | Water Intensity Index |
|---|---|---|
| A | 0.40 | 0.22 |
| B | 0.40 | 0.42 |
| C | 0.40 | 0.17 |

As seen in Table 8, all designs have similar mass intensity index. This is because the design at point A, point B and point C used equal feed flowrate and produce the same product flowrate. By comparing the water intensity index for each point, it can be seen that point C has lower index compared with other points followed by Point A and Point B. This is due to the lowest usage of water in the process of distillation. Although design at Point A is not the best in term of material consumption, but it is still not the worst among the all the designs. Besides, other criteria also need to be considered and the best design will be decided in Stage 6.

*Step 4.2: Energy Consumption*

Besides material consumption, energy consumption in each process also plays a major role to the sustainability assessment [12]. This is due to the excessive usage of energy will leads to economic losses and negative environment impact. By calculating energy intensity, the energy impact of the separation Styrene – p-Xylene based on each point of design is presented in the Table 9.

**Table 9:** The values of energy intensity at different distillation designs for styrene – p-xylene separation system.

| Distillation Design | Energy Intensity Index |
|---|---|
| A | 0.30 |
| B | 0.52 |
| C | 0.24 |

Table 9 shows that design at Point C has the lowest of energy consumption compared to other designs. From Table 8 and Table 9, its shows design at Point B is the worst of sustainable design. Point A also not the best design for good energy consumption. However, it still needs to consider others criteria and the best design will be decided at stage 6.

***Stage 5: Detailed Economic Analysis***

The objective of this analysis is to identify and evaluate the economic in order to proof that by designing using driving force concept, it will result in the maximum profit. To proof that, the distillation design will be analyzed by using the profit function.

*Step 5.1: Profit Function*

The economic analysis of each distillation designs (Points A, B, C) is analyzed by calculating it using the profit function. There are four criteria that must be calculate in order to achieve profit function which are income of the product, cost of raw material, depreciation cost (equipment cost) and operation cost (water and electricity usage) [12]. The economic analysis is calculated by assuming that the distillation column is running in 5 years, and based on 340 days of operation each year. The result is tabulated in Table 10 in U.S Dollar ($).

**Table 10:** The values of income of product, cost of raw material, depreciation cost, operating cost and profit function, Ø at different distillation design for styrene – p-xylene separation system.

| Distillation Design | Product Cost ($) | Raw Material Cost ($) | Operation Cost ($) | Depreciation Cost ($) | Ø ($) |
|---|---|---|---|---|---|
| A | 241,122,442 | 20,573,808 | 814,515 | 4,495,850 | 215,238,269 |
| B | 241,122,442 | 20,573,808 | 844,773 | 7,850,378 | 211,853,484 |
| C | 241,122,442 | 20,573,808 | 804,717 | 3,508,828 | 216,235,090 |

From the results tabulated in Table 10, it is seen that the distillation design at point C has the highest profit function compared with design at point A and Point B. This is due to distillation design at point C has the lowest depreciation cost (equipment cost) and lower operating cost compared to other design. Later, the best distillation design will be verified in stage 6 using multi objective functions to find the best candidates of distillation design.

***Stage 6: Final Selection and Verification***

The objective of this stage is to select the best candidate by analyzing the value of multi objective function, Eq. 14. The highest value of $J$ is selected as the best distillation design.

*Step 6.1: Final Selection: Verification of Design*

In this step, the multi objective function is calculated using Eq. (14) by summing up the multi objective function value. For this case, the decision maker will not have any preference for one objective with another as the weight objective function is weighted equally. Each objective value is normalized with respect to the highest value since the range of the objective function can be different. As shown in Table 11, $P_1$ is the scaled value of the driving force $F_{Di}$, $P_{2,1}$ and $P_{2,2}$ is the values of $dF_{Di}/dx_{X\text{-}DE}$ and $dx_{B,S}/dL$ which is representing the value of sensitivity analysis of driving force $F_{Di}$ with respect to $x_{X\text{-}DE}$ as a disturbances and derivatives value of bottom column composition $X_{B,S}$ with respect to step down of $L$ , respectively. $P_{3,1}$ and $P_{3,2}$ are the scaled value of material consumption and energy consumption in the

sustainability analysis. Lastly, $P_{4,1}$ is the scaled value of profit function, Ø at different design points in the distillation column.

**Table 11:** Multi-objective function calculation. The best candidate is in bold.

| **Distillation Design** | $P_1$ | $P_{2,1}$ | $P_{2,2}$ | $P_{3,1}$ | $P_{3,2}$ | $P_4$ | |
|---|---|---|---|---|---|---|---|
| A | 0.4295 | 0.1036 | 0.3212 | 0.62 | 0.30 | 215,238,269 | |
| B | 0.3112 | 1.9446 | 0.1318 | 0.81 | 0.52 | 211,853,484 | |
| C | 0.3872 | 0.4268 | 0.5008 | 0.57 | 0.24 | 216,235,090 | |
| | $P_{1s}$ | $P_{2,1s}$ | $P_{2,2s}$ | $P_{3,1s}$ | $P_{3,2s}$ | $P_{4s}$ | $J$ |
| A | 1.0000 | 0.0533 | 0.6413 | 0.7594 | 0.5819 | 0.9954 | **24.43** |
| B | 0.7245 | 1.0000 | 0.2632 | 1.0000 | 1.0000 | 0.9797 | 4.97 |
| C | 0.9015 | 0.2195 | 1.0000 | 0.6969 | 0.4554 | 1.0000 | 11.09 |

So, it can be seen that from the table the multi objective-function $J$ for an extractive distillation column design A is higher compare to other designs. Therefore, it verified that, extractive distillation column for design A is the optimal solution for sustainable integrated process design and control (*Sustain*-IPDC) of a styrene – p-xylene separation process which satisfies the design, control, sustainability and economic criteria. It should be noted that a qualitative analysis ($J$ highest for point A) is sufficient for the purpose of control structure, sustainability and net profit selection, but it is not the worse design in those criteria. This make the distillation design at point A is the optimal design.

## 4.0 CONCLUSION

In this paper, a systematic model-based methodology has been developed for sustainable integrated process design and control (*Sustain*-IPDC) for an extractive distillation column system. Here, the *Sustain*-IPDC problem for an extractive distillation column system is solved by decomposing it into six sequential hierarchical sub-problems: (i) pre-analysis, (ii) design analysis, (iii) controller design analysis, (iv) sustainability analysis, (v) detail economics analysis, and (vi) final selection and verification. It should be noted that sub-

problems (iv) and (v) are the new sub-problems added to the previous developed methodology to undertake the sustainability and detailed economic analysis. The developed *Sustain*-IPDC methodology was able to find the best solution which satisfied design, control, sustainability and economic criteria in efficient and systematic manner through the separation of Styrene – p-Xylene mixture.

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