INTENSIFIED FLEXIBLE DISTILLATION PROCESS FOR MULTI-PERIOD OPERATION

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ABSTRACT

Canada's aspirations towards an energy independent future mean that it can ill-afford to rest while the rest of the world pursues sustainable energy technologies and other efficient means of energy and chemical fuels production. Fortunately, the abundance of biomass—a sustainable energy resource—within Canada's borders provides a great opportunity for those seeking to help mitigate the threat of fossil fuel induced climate change to intervene; by taking advantage of this abundant resource and using it in new and improved energy and chemicals production pathways. Our current work, therefore, derives from this notion; and in it, our objective is: the development, design, and optimization of a novel separation process for producing high purity—chemical grade—Methanol (MeOH) and Dimethyl Ether (DME) from biomass-derived syngas.

Approaches surveyed often focus on designs of entirely new pieces of equipment, but our work is a take on subtly but critically improving one of the oldest and most ubiquitous pieces of process equipment: tray distillation columns. Using a novel approach for solving the optimization problem, an algorithm is furnished and this is implemented in Matlab, while all process simulations are performed in Aspen Plus. The development of a rigorous framework for designing intensified flexible distillation process for high purity DME-MeOH production is one main contribution of this work. Another major finding is that an intensified flexible process design for DME-MeOH separation for multi-period operation has lower total annualized cost compared the conventional processes. Furthermore, the proposed process shows a lower penalty for being flexible when compared with the status quo. Although further studies on the effects of process dynamic behaviour, including on transitions and other transient characteristics, are needed, the findings so far suggest a potential for substantial life-time cost savings in new process designs for DME-MeOH separations.

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LIST OF ABBREVIATIONS AND SYMBOLS

Abbreviation	**Description**	**Units**
DME	Dimethyl Ether	-
MeOH	Methanol	-
DOM	DME Operating Mode	-
MOM	MeOH operating Mode	
H_2O	Water	-
x_{DME}	Mole fraction of dimethyl ether	-
x_{MeOH}	Mole fraction of methanol	-
x_{H2O}	Mole fraction of water	-
RR	Reflux ratio	-
BR	Boil-up ratio	-
TAC	Total Annualized Cost	$/yr
TDC	Total Direct Cost	$
UTL	Total Annual Utility Cost	$/yr
$Cost_{Ann.Cap}$	Annualized capital cost	$/yr
N_S	Total number of stages	-
N_F	Location of feed line (above stage)	-
N_A	Number of stages above the feed line	-
N_B	Number of stages below the feed line	-
D	Diameter of distillation column	ft.
A_C	Condenser [Surface] Area	m^2
A_R	Reboiler [Surface] Area	m^2
$Cost_C$	Condenser purchase cost	$
$Cost_R$	Reboiler purchase cost	$
$Cost_{dc}$	Distillation column purchase cost	$
$Cost_{QC}$	Cost of condenser utilities per hour	$/hr
$Cost_{QR}$	Cost of reboiler utilities per hour	$/hr
MPS	Medium Pressure Steam	-
REFRIG	Refrigerant	-
Q_C	Condenser heat duty (J/s)	watt
Q_R	Reboiler heat duty (J/s)	watt
C_{CU}	Cost of cold utility ($/GJ)	$/GJ
C_{HU}	Cost of hot utility ($/GJ)	$/GJ
ϕ	Fractional amount of time in DME mode	-

Chapter 1 INTRODUCTION

1.1 Motivation

Abundance of coal, biomass, natural gas, and petroleum sources globally has incentivized the production of energy products, such as liquid fuels, power, and chemicals from these carbonaceous feed stocks. In Canada the feed stocks with the greatest potential that are being considered are biomass and natural gas, with Canada currently ranked 5th and 7th, respectively for global natural gas production and consumption (BP, 2016). Due to prevailing and/or looming economic and socio-political barriers, like carbon taxes, increasingly stringent environmental measures enacted to limit carbon dioxide emissions (such as, the Paris Agreement), it is becoming progressively challenging to meet the planet's energy needs using fossil resources in traditional processes, such as in pulverized coal power generation. Instead, novel processes that produce energy while limiting the amount of CO2 released into the atmosphere are needed. The most promising among these innovative technologies is gasification, from which synthesis gas (syngas)—a mixture comprising mainly carbon monoxide and hydrogen—is derived. Syngas is a feedstock in many chemical synthesis and power generation processes, most notably in Dimethyl Ether (DME) Synthesis.

DME—a colorless, odorless gas at standard conditions—is a versatile compound that can be used in many chemicals application areas such as, as an alternative (synthetic) fuel in diesel combustion-ignition engines, due to DME having a lower auto-ignition temperature and a higher cetane number than diesel (Fleisch et al., 1995). DME also has use as: a solvent and an aerosol, as a hydrogen source in fuel cells, as fuel in gas turbine power generation (Azizi, Rezaeimanesh, Tohidian, & Rahimpour, 2014), and as feedstock in olefins production (Sardesai & Lee, 1998) among others. DME is nearly as versatile as syngas, while offering increased utility in that it is easier to store, handle, transport and distribute compared to syngas: DME is readily condensed to a liquid a relatively low pressures of just over 5 bar (Arcoumanis, Bae, Crookes, & Kinoshita, 2008) . This true versatility is further bolstered by the fact that DME, the simplest of the aliphatic ethers, has a particularly simple structure which affords it many of the advantages it possesses over other chemicals. Since DME lacks a carbon-carbon bond, present in many fuels, and also has higher O_2 content, it burns very cleanly; releasing very little smoke or particulate matter (Fleisch et al., 1995), and low NOx and CO emissions (Azizi et al., 2014).

Large-scale production of DME is achieved via dehydration of methanol (MeOH), usually on a, a dehydration catalyst such as γ-Al_2O_3, an acid catalyst in a so-called “indirect” process (Bercic & Levec, 1993), (Xu, Lunsford, Goodman, & Bhattacharyya, 1997),(Müller & Hübsch, 2000). Dehyndration can also be achieved with the use of a copper-zinc-oxide catalyst (Wender, 1996). Although it has not yet found wide industrial adoption, the synthesis of DME directly from syngas in a so-called “direct” method nevertheless has been widely demonstrated (Brown, Bhatt, Hsiung, Lewnard, & Waller, 1991), (Ogawa, Inoue, Shikada, & Ohno, 2003). The majority of MeOH synthesis at commercial scale uses synthesis gas (syngas) derived from natural gas reforming (Kim Aasberg-Petersen et al., 2013; Ott et al., 2011), although some other carbonaceous feedstocks such as biomass and coal can also be used as a raw material via a gasification process (Larson, Jin, & Celik, 2009). The main components of the crude methanol coming from the synthesis reactor are MeOH, H_2O, and traces of DME and EtOH (Clausen, Houbak, & Elmegaard, 2010).

DME production is intricately related with MeOH production, and as such, any attempt at improving DME process performance and/or economics necessarily has to also examine the role of MeOH. The direct synthesis route produces a mixture of DME which comprises high levels of dissolved $CO_{2,}$ requiring highly complex and costly separation (Pascall & Adams, 2013). In contrast, in the indirect method for DME synthesis, the reactor effluent from the MeOH dehydration comprises a simple ternary mixture of MeOH, DME, and water, which requires comparatively low cost of separation. Because the indirect DME synthesis route is the most widely used in industry, any improvement of process economics in this design has a high impact potential. Motivated by this reasoning, a process of optimization of the separation process for the distillation of DME and MeOH is undertaken. The optimization implementation envisaged involves the reduction of separation equipment footprint by applying process intensification. By repurposing distillation equipment—using the same distillation columns for the separation of two different feeds, at two different time periods—substantial capital and operating cost savings can be had. Compared with the conventional distillation sequences, the proposed “flexible” distillation design strategy will improve profitability of the DME-MeOH distillation process.

1.2 Background

The DME-MeOH sections considered are those belonging to a polygeneration plant shown in **Figure 1.1.** Polygeneration is a process in which a portfolio of energy products is made, possibly at various times in a given operating day, from one or more energy feedstocks. Polygeneration's chief advantage is that, by design, it is more flexible than single product plants. Polygeneration allows for the exploitation of inherent synergies between chemicals production and power generation. This portfolio optimization strategy not only mitigates economic risk{by guaranteeing that the most profitable products are produced at all times{but also maximizes profit made from the sale of products within the portfolio (Adams & Barton, 2011).

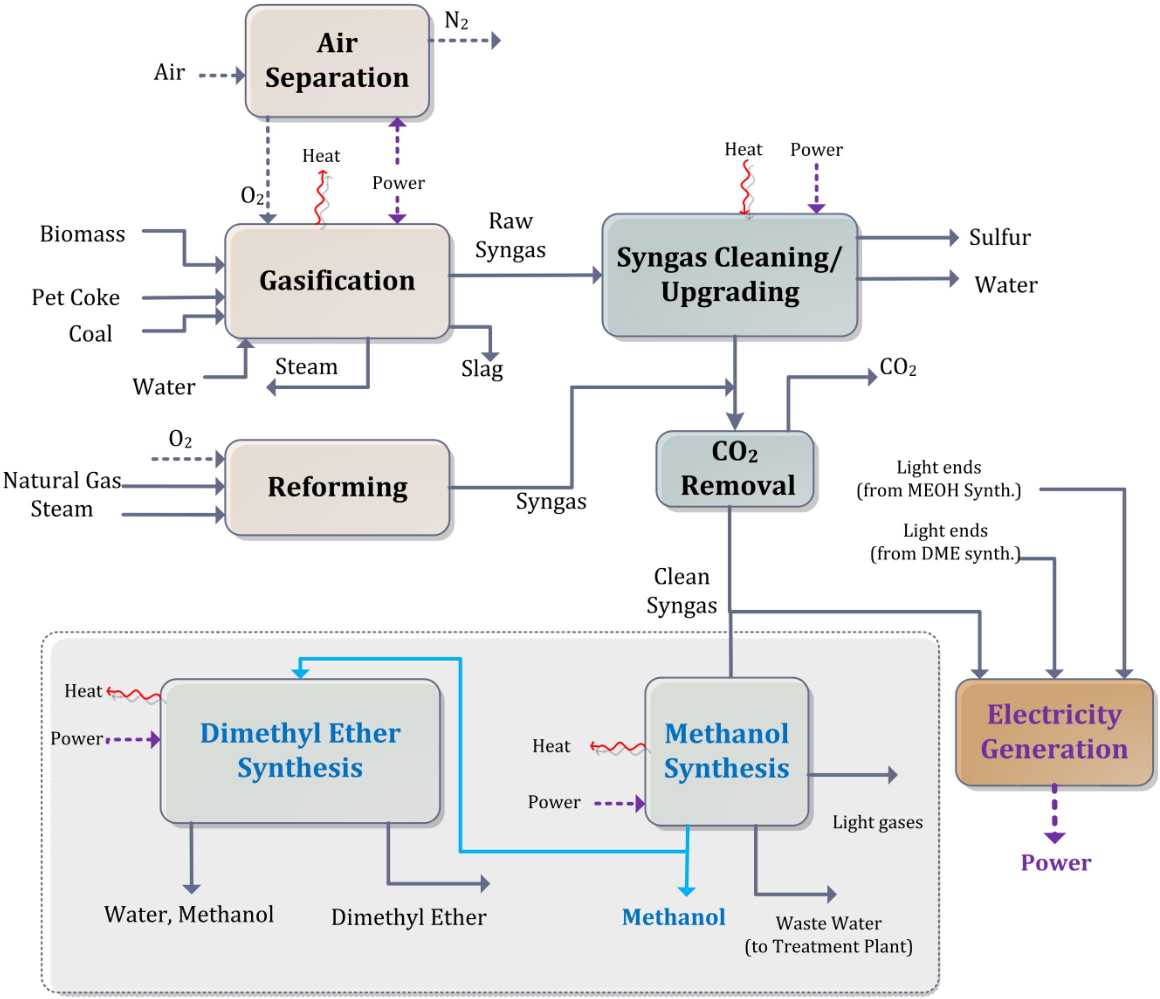

Figure 1.1 Overview of a polygeneration process for synthetic fuels and electricity production (Adams & Barton, 2011)**. DME and MeOH synthesis sections are highlighted.**

The separation of MeOH through a conventional industrial process involves continuous distillation sequences, often employing two—but sometimes more—distillation columns in series (Ott et al., 2011), (Cheng & Kung, 1994). The first column is a topping column in which light ends are removed, while the second is a refining column in which waste water and heavy ends are removed, with pure methanol collected separately as the distillate (Ott et al., 2011). Similarly, conventional dimethyl ether (DME) separation is accomplished via a two-column train (Müller & Hübsch, 2000).

1.3 Objectives

The aim of this research is to design a *multi-purpose* two- column distillation process for the separation of chemical grade DME (99.95 wt. %) and chemical grade MeOH (99.85 wt. %). A design configuration--including equipment sizes—that is both flexible and optimal under various market scenarios is desired. The merits of this proposed design will be compared and contrasted to the two *parallel* trains, each with a sequence of two distillation columns, currently employed for the conventional separation of DME and MeOH. The study will be on an industrial process producing up to 100 kilo tonnes DME/year.

The design envisaged should be capable of switching between two feeds (of non-azeotropic mixtures) coming from different sections of the plant (**Figure 1.1**)—the MeOH synthesis and DME synthesis section--with rather distinct compositions and flows, and at two different points in time. The simulation of all process designs is to be carried out in the Aspen Plus (“Aspen Plus,” 2015) environment. The following steps will be followed in order to meet the stated objective:

i. Simulation of the conventional four column distillation sequence, of two parallel trains to achieve specified product purities for DME and MeOH.
ii. Optimization of the conventional design process

iii. Optimization of the proposed flexible distillation sequence comprising a single train of two columns.
iv. Comparative analysis of the economics of the proposed flexible design against the conventional design

These chemical grade purities specifications on the products are crucial in order for the process to be economically viable, as the products will be sold for revenue. Any designs which fail to meet these product specifications are not considered realizable and are thus discarded. The proposed distillation design ought to demonstrate measurable improvement over the two current continuous cases. Therefore, a total annualized cost (TAC) analysis criterion is used in order to assess the potential benefits of the proposed process intensification scheme. For the proposed scheme to warrant adoption, it has to be of at most equal TAC as the status quo, otherwise it is economically infeasible.

1.4 Main Contributions

A process intensification strategy, in which a flexible separation process comprising a single train of two distillation columns (in place of a conventional two-train setup), is developed and optimized for the production of high purity DME and MeOH. The final result is a distillation column sequence capable of meeting product specifications for DME and MeOH purities, while possessing the ability to switch between two different feeds, and at two different points in time.

Thus the proposed, optimal design has the following attractive attributes: two fewer distillation columns (two versus four) compared to the two original continuous cases, lower overall capital cost, and consequently lower total annualized cost (TAC). In addition, the proposed design will have improved flexibility in responding to changing market conditions.

Last but not least, the optimization problem being tackled is highly nonlinear, nonconvex, and multimodal. To better tackle these nuanced complexities, the help of process modeling software capable of providing high fidelity results is employed. It is hoped that the findings from this work can be applied to designing of processes for retrofitting existing DME-MeOH plants, or for implementation in future designs of those types of plants.

Further, the methodology developed can be readily extended to similar other application areas, such as in Fischer Tropsch process for liquid fuels production.

1.5 Thesis Outline

The remainder of this thesis is arranged as follows:

Chapter 2 – Literature Review

Through the surveying of at relevant literature, prior and ongoing efforts in the areas of distillation process intensification for DME separation applications and process flexibility are discussed.

Chapter 3 – Process Modeling and Optimization

This section presents the approach followed in solving the modeling and optimization problem stated. Aspen Plus and MATLAB (“MATLAB,” 2014) are employed in to achieve this.

Chapter 4 – Modeling and Optimization Results

This section presents the results obtained from the modeling and optimization of both the conventional four-column distillation and the proposed flexible two-column processes. Comparative economic assessment of the two process designs is undertaken.

Chapter 5 – Conclusions and Recommendations

This section summarises the main findings as stated in the objective. Main contributions of the results are also highlighted. Finally, recommendations on future studies are discussed.

Chapter 2 LITERATURE REVIEW

Optimal design of distillation columns is often a challenging endeavour due to the problem being a mixed-integer non-linear programing (MINLP) problem. The design of a *flexible* distillation column is no different, requiring solution of discrete (number of stages, feed stage) and continuous (internal ratios, product purities) variables. This is further compounded by the fact the problem structure for flexible designs is often unknown, and by definition possesses numerous non-convexities and nonlinearities, and even multi-modality. Conventional approaches which employ mathematical (gradient-based) optimization techniques often suffer from poor and premature convergence to locally [sub] optimal solutions; even globally oriented algorithms employing exact methods often require enormous runtimes—which increase in proportion to the number of integer variables in the MINLP—to yield globally optimal solutions. In addition, these techniques are highly complex and require considerable effort—both human and computer—for their successful implementation. Further, effective solution of such MINLP problems requires good initial guesses, which can be difficult to obtain As a result of these complications, many researchers resort to employing shortcut methods or reduced order methods--in place of full complex ones in order to make the MINLP problem tractable-- when solving flexible design problems, leading to results which have limited application; with little to no direct industrial application. Clearly, an approach that preserves model detail and complexity can potentially yield higher fidelity results and would thus be preferable to methods that use reduced-order process models to solve the problem of optimal design of flexible distillation columns. Employing the full power of rigorous process simulation software such as, Aspen Plus, wherever possible, could prove more advantageous over other optimization techniques which employ full Mathematical rigor but sacrifice model fidelity. Rigorous simulation methods can produce highly accurate results as a result of built-in property packages and heuristics derived from real-world performance of designed plants and processes. This point is illustrated by a survey of several works from the open literature.

2.1 Flexibility Analysis and Design under Uncertainty

Grossman and colleagues (I.E. Grossmann, Halemane, & Swaney, 1983) posit that there are in fact two classes of problems when considering the design of chemical processes for feasible steady-stage operation under parametric uncertainty. The first class of is that of *design for multi-period operation*—designing chemical processes that are able to operate optimally in successive time periods under disparate operating requirements. The second class of problems falls under the area of *design under uncertainty*. Although other complex design problems can be a combination of the two classes (Chien, Douglas, & Penlidis, 1991). Algorithms are presented for solving both classes of problems. Tellingly however, due to the complexity and large size of this first class of problems, the researchers assume fixed length of time in each nth time periods and solve a restricted version of the multi-period design problem. Our variable period of operation necessarily makes our problem different from the usual design under uncertainty problems just discussed.

2.1.1 Flexible Chemical Plants for Multi-Period Operation

Grossmann and Sargent (Ignacio E. Grossmann & Sargent, 1979) consider the design of chemical plants for operating in different time periods. In each period the conditions are constant, but between periods the conditions are allowed to vary, leading to a piecewise constant operating regime. Switching costs are not accounted for as they are assumed to be negligible if the length of each operating period is sufficiently long. The methodology used assumes that the objective function is differentiable, thus leading to a NLP. An additional examples involving batch process is also given, whose problem formulation leads to an MINLP, but this is not solved to optimality however.

Varvarezos and colleagues (Varvarezos, Grossmann, & Biegler, 1992) propose using an outer approximation algorithm to solve the difficult MINLP resulting from the formulation of design problem for chemical plants operating in multiple successive periods. The need for employing decomposition strategies is stressed as these multi-period design problem have a large number of variables and constraints, which can often make the problem intractable.

2.1.2 Design under Uncertainty

For the design under uncertainty, Grossman and colleagues (I.E. Grossmann et al., 1983) consider both the case when the parametric uncertainty is fixed and specified and the more complex case when uncertainty is unknown but bounded. When the uncertainty is unknown, the objective is to optimize the degree of flexibility of the process under design. This leads to the need for quantification of flexibility, which the researchers accomplish using flexibility test—the extent of flexibility of a design-- and a flexibility index—a numerical value that measures scaled deviations from operation that the design can tolerate given the uncertainty in the parameters--formulation. The smallest overdesign is sought which assures feasible operation at minimum cost.

Swaney and Grossmann (Swaney & Grossmann, 1985) address in more detail the Mathematical underpinnings of the flexibility index developed earlier by Crossman and colleagues. The design under uncertainty culminates in the classic "Max-Min-Max" feasibility problem (with objective function being any form of a cost function), subject to this "Max-Min-Max" constraint. The "Max-Min-Max" constraint is involves: determining the smallest maximum constraint violation tolerable while still guaranteeing feasible operation over the greatest uncertain parameter space.

Grossmann and Floudas (I. Grossmann & Floudas, 1987) develop efficient methods for addressing an earlier problem presented by the max-min-max optimization problem first postulated by Swaney and Grossman (Swaney & Grossmann, 1985) which could not be solved by standard NLP methods due to the feasibility constraint being non-differentiable. By identifying groups of constraints that limit the flexibility of a design (that is, active constraints), the researchers are able to recast the problem as an MINLP and use an active-set strategy to solve this now tractable problem.

Pistikopoulos and Grossmann (Pistikopoulos & Grossmann, 1989) extend the work earlier work of Grossman and collaborators (I.E. Grossmann et al., 1983), (I. Grossmann

& Floudas, 1987) to include the case of retrofit designs. In the retrofit design, design alterations on existing equipment are sought which will yield a more flexible process while ensuring the operation of the process remains feasible under expected parametric uncertainty.

Kabatek and Swaney (Kabatek & Swaney, 1992) proposed an improved branch and bound strategy for solving the design under uncertainty problem. In essence, their technique improves the identification of active constraints—corresponding to the worst-case uncertainty realization that limits process flexibility.

Pistikopoulos and Mazzuchi (Pistikopoulos & Mazzuchi, 1990) extend the work of Pistikopoulos and Grossmann (Pistikopoulos & Grossmann, 1989) to account for cases when the uncertainty is not deterministic but is instead random (and continuous). Observing that parametric uncertainty is often unknown, they argue that its modelling should be stochastic. The uncertain parameter space is represented by a probability distribution. The investigators further reason that this approach allows for accurate modelling of the parametric uncertainty, including for instances when the different parameters are highly correlated. However, their methodology is only applicable to linear systems. Their findings lead to a new paradigm in design under uncertainty; that of “stochastic flexibility.” Unlike earlier deterministic formulations, this gives the *probability* that a design will remain feasible during operation given the expected parametric uncertainty. The objective is to maximize this probability.

To address the limitations of the linear model assumptions in previous work by Pistikopoulos and Mazzuchi (Pistikopoulos & Mazzuchi, 1990), Straub and Grossmann (Straub & Grossmann, 1993) propose a strategy for modeling stochastic uncertainty during the design of nonlinear processes. The resulting NLP is solved using standard NLP algorithms. The researchers' approach also allows for generation of relational curves which enable the evaluation of trade-off between process flexibility and cost. For an overall overview of design of chemical plants under uncertainty, readers are directed toward the work of Morari (Morari, 1983) Grossman et al. (Ignacio E. Grossmann, Calfa, & Garcia-Herreros, 2014).

2.2 Flexible[1] Distillation Column Design

Distillation is said to account for approximately 90% of all separation processes used in the chemical process industries (Haan & Bosch, 2015, p. 333). In this subsection all literature sources reviewed are concerned specifically with distillation process, as is the focus of our own research. Extensive research in the area of flexible distillation design is focused on the general *optimal synthesis* problem—the determination of the optimal sequence of distillation column required for a given separation. In many synthesis problems, since the focus is on determining the optimal sequence, simple distillation models are often favoured in place of rigorous ones. Limited research exists in the area of design of distillation columns for flexible operation.

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[1] A flexible/multipurpose distillation plant is described here as one that is capable of separating more than one kind of feed with the same equipment.

Wagler and Douglas (Wagler & Douglas, 1988) propose a simple method termed “Practical Feasible Analysis,” which is used in synthesis (choosing difference sequences) of flexible distillation column sequences under uncertainty. The methodology relies on extensive process knowledge and heuristics on the part of the design engineer, limiting its application. Moreover, the authors use simple Fenske-Underwood-Gilliland methods for modeling the distillation process. These short-cut methods are quite simplistic and are accurate only for ideal component mixtures. In addition, very simplistic assumptions are made on critical distillation column performance parameters and during capital cost estimation.

Paules and Floudas (Paules & Floudas, 1988) propose an MILP formulation for identifying flexible distillation sequences under multi-period operation *under N discrete feed compositions.* The length/duration of each period is assumed known and is fixed. The work addresses the synthesis problem of distillation sequences that have the flexibility to handle known changes in the composition of the multicomponent feed stream for a finite number of periods of operation.

The researches’ approach is a substantial improvement on Wagler and Douglas (Wagler & Douglas, 1988)’s simplistic approach that only used simple shortcut methods. In addition, this work goes on to address the case of different feeds, not just feeds in which component concentration vary somewhat. The inclusion of rigorous process models (simulated in Aspen Plus (“Aspen Plus,” 2015)) is also a significant improvement. The main limitation, however, is that the number of stages (N_S) and feed tray location (N_F) are not optimization variables, instead a nominal column is used whose N_S and N_F values are the result of shortcut methods. Simpler models (fixed-charge models) are derived from the simulation results and these form the basis of the optimization (cost minimization). Later work by Paules IV and Floudas (Paules & Floudas, 1992) extend on this work further and investigates case of stochastic uncertainty of the synthesis problem for multi-period operation. The resulting problem is a complicated and difficult two-stage stochastic MINLP problem.

Huss and Westerberg(R S Huss & Westerberg, 1996; Robert S. Huss & Westerberg, 1996) use collocation models for distillation columns. The formulation relies on the transformation of the number of stages (N_S) into a continuous variable; an approximation of the slope of the polynomial over the column section is taken as the number of stages. In addition, the reflux ratio (RR) is assumed as a simple function of number of stages, N_S.

The collocation approach uses approximation of discrete variables (namely number of stages above and below feed) into continuous ones so that the problem just reduces to an NLP, the solution of which is relatively straightforward and can be achieved with standard NLP algorithms. This was done intentionally to circumvent the problems that arise from solving then mathematically intractable MINLPs as mentioned earlier. However, later development of efficient techniques for solving this problems coupled with the increased use of process simulators for solving distillation problems provided a better alternatives, the latter of which provides highly accurate results.

The researchers’ approach does not permit use of a simulation package. In addition, the resulting collocation model can have many inaccuracies due to many approximations, resulting in convergence of the model on solutions that are physically unrealizable. The researchers also note that using product purities as degrees of freedom results in problems failing to converge. Otherwise, the researchers note that certain "tricks" such as, choice of which components to specify, end up being necessary in order to make problems converge. This limits the application of the collocation approach to a select number of separation problems.

Chien et al (Chien et al., 1991) extend the earlier work of Wagler and Douglas (Wagler & Douglas, 1988)’s Practical Feasibility Analysis approach, adding the flexibility index calculation of Grossman and Swaney (Swaney & Grossmann, 1985) to determine precisely the degree of flexibility of a proposed design. Other improvements include replacing short-cut methods with rigorous models (in Aspen Plus).The proposed methodology addresses the complex “multi-period design of a flexible process under uncertainty” problem. However, the numbers of stages above and below the feed are not rigorously optimized; instead they remain at their nominal values such that the resulting problem is an NLP. A standard NLP algorithm (Augmented Lagrangian) is then used to solve the optimization problem.

In this section**,** overwhelmingly, simple or reduced order models are used throughout since most of the literature is focused on mathematical techniques. This motivates the discussion involving rigorous process models, implemented in process simulation software. Process simulators have improved accuracy due to the use of detailed property packages that allow for accurately determining thermodynamic and transport properties of compounds (in contrast to simple methods which use simplified mass and energy balances).The section that follows presents work in which process simulation tools are vital for the design of multipurpose plants.

2.3 Process Intensification Techniques in DME-MeOH separation

Buoyed by an ever increasing use of highly capable process simulation tools in chemical plant design research, recent work on enhancing plant economics through novel design has focused more on techniques which employ a process intensification philosophy. This is especially true for distillation processes. Stankiewicz and Moulijn (Stankiewicz & Moulijn, 2000) define process intensification as any undertaking during chemical process design which results in significantly smaller, cleaner, and more efficient designs or processes.

Examples of this design philosophy from literature are taken from the processes involving the distillation of DME-MeOH; all rigorous modelling is done in a process simulator:

- Semicontinuous Distillation:

  Pascall and Adams (Pascall & Adams, 2014) perform a dynamic simulation of a semi-continuous distillation process for the separation of a multicomponent mixture of water, MeOH, and DME, and CO_2. The researchers also perform a detailed optimization of the dynamically operating column, including the design of appropriate controllers. An economic comparison of the semi-continuous distillation design with the conventional distillation process is also made.

- Dividing Wall Column

  Kiss (A. A. Kiss, 2013) presents work on the use of dividing-wall columns (DWC) for the separation of multicomponent feed stream from biofuels processes, including MeOH and DME purification. Various versions of DWC implementation are also provided, with extensive application areas such as, azeotropic separations. Economic benefits of the different DWC variants compared to conventional designs are also highlighted. A more detailed review of DWC technology can be found in work by Dejanovic et al (Dejanovi, Matija Sevi, Jansen, & Oluji, 2011).

- Reactive Distillation / Catalytic Distillation

  An et al. (An, Chuang, & Sanger, 2004) propose a reactive distillation (RD) design in which MeOH is catalytically dehydrated into DME and water in a single reactor-separator unit. Kinetics of the rigorous reaction and distillation models are evaluated. The potential for substantial capital cost reduction compared to the conventional process are highlighted, following optimization of the various distillation unit's design and operating parameters.

- Reactive Distillation + Dividing wall column

Kiss and Suszwalak (A. A. Kiss & Suszwalak, 2012) present a design which reputedly combines the benefits of DWC and RD into a single unit. The resulting design is termed a reactive dividing wall column (R-DWC). The economic performance of the optimal design process for the separation of a ternary mixture of water, MeOH, and DME is compared against the conventional design process.

It is important to note that while all the above process intensification strategies have been attempted in the past, our proposed approach is different and unique. The main difference is that all four approaches discussed above are technologies have not yet found wide industrial use, or have very specific applications[2]; conversely, our approach takes a conventional distillation column and uses it in a totally new way that, in our estimation, has never before been investigated.

2.4 Summary

Process flexibility has been studied extensively by various researchers over the years. Most earlier works focus on mathematical formulation of flexible design under uncertainty and the development of solution techniques for tackling the resulting complex optimization problems. Later approaches include process intensification strategies and increased deployment of process simulators to obtain rigorous and more accurate results. Clearly, approaches which can combine flexibility analysis *and* process intensification—all implemented in a powerful process simulator—may have greater utility and therefore may be more attractive. In this work, we attempt to design such a process: an optimal

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[2] Semicontinuous distillation is applicable in small-scale separations, becoming prohibitively expensive with increasing scale.

design that is not only flexible under multi-period operation but also has a reduced footprint. Uncertainty is in the duration of each period of operation. However, quantification of flexibility, as in the manner of Swaney and Grossman (Swaney & Grossmann, 1985), is beyond the scope of this work. The implementation of our proposed strategy will be detailed in the chapters that follow.

Chapter 3 METHODOLOGY FOR FLEXIBLE DISTILLATION COLUMN DESIGN FOR DME/MEOH SEPARATION

3.1 Introduction

Crude mixtures from the two synthesis processes for DME and MeOH both comprise the same three main components, namely, DME, MeOH and H_2O[3]. Thus our goal is to be able to have both crude feeds be, with some success, able to be sent to the same sequence of (two) distillation columns to achieve the desired separation of the three main constituents at high purity, with some careful adjustments to process operating conditions during the two distinct modes of operation..

The task at hand then is to design a separation (distillation) sequence which is capable of accepting the two very different feeds—one from the MeOH synthesis process and the other from DME synthesis. The design envisioned necessarily has to be able to achieve separation purities of 99.95 wt.% and 99.85 wt.%, respectively for chemical grade DME (Müller & Hübsch, 2000) and chemical grade MeOH Furthermore, during the simulation phase of the process design, the presence (in parts per million) of other reaction by-products—such as acetone, ethanol, methyl formate, and some dissolved gases—in the crude MeOH feeds is deprioritized, as the three main constituents—DME, MeOH, and H_2O—of the feed streams have the greatest impact on process design criteria, while the other components, which are present only in extremely minute (ppm) concentrations have negligible influence.

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[3] There is also some unreacted syngas in the synthesis reactors' effluents, which are typically removed by flash columns prior to entering the distillation section of the plant.

A total annualized cost (TAC) analysis criterion is used in order to assess the potential economic benefits of the proposed flexible process intensification scheme.. Not only does the proposed design need to demonstrate economic feasibility but it also has to be technically feasible. Process simulation is used to address the technical feasibility problem while process optimization is used to steer the feasible design towards the most favourable economic performance.

The sections that follow in this chapter underline the methodology followed in the design of the two—the conventional and the proposed—processes. First, the modelling approach used for the processes is laid out, followed by a detailed description of the simulation phase. Lastly, the crux of the optimization approach--is used to assess the processes ‘economic performance--is detailed.

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3.2 Process Modelling

3.2.1 Conventional Design Case: DME and MeOH Purification

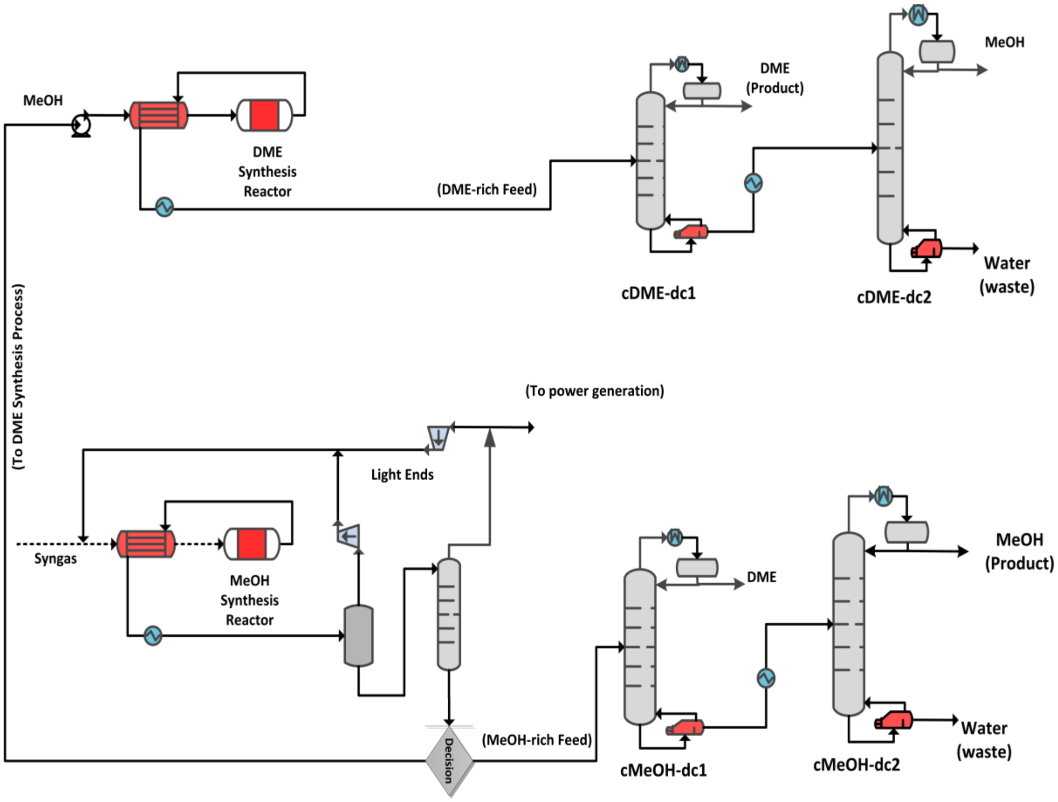

Figure 3.1. Conventional two-column distillation sequence for purification of dimethyl ether (top) and methanol (bottom).

The conventional process for DME production from MeOH is shown in the top section of **Figure 3.1**. Crude methanol first enters the DME synthesis reactor, where the MeOH is catalytically transformed into DME and water. The reactor effluent, which is made up of DME, water, and unreacted MeOH then goes to the distillation section. In the first distillation column, a high pressure column at 10 bar, the DME is removed at a high purity (99.95 wt. %) at the top, while in the second column the unreacted methanol is enriched and removed at high purity at the top. The enriched MeOH can be sold for revenue or recycled back to the DME synthesis reactor. For the purposes of our investigation, the case of recycled MeOH is not being considered, instead the high purity MeOH stream (99.85 wt. %) is to be removed as product and sold. The waste water, which will often be sent to a water treatment facility, is recovered as a high purity (>99.9 wt. %) stream in the bottoms of the second column. Due to DME's low boiling point at the operating conditions in the first column, the use of a refrigerant as the condenser utility is required[4]. But since the methanol distillate has a relatively high boiling point at the chosen operating conditions, the use of cooling water as the condenser utility is permissible in the second column. On the other hand, the presence of considerable amounts of water—a higher boiling component than either DME or MeOH—in the bottoms of both columns necessities the use of medium pressure stream as the utility for the reboiler. The column pressure was not selected as an optimization variable for the purpose of this work; appropriate values for column pressures are adapted from the open literature (A. A. Kiss & Ignat, 2013) for an identical process.

The bottom half of Figure 3.1 illustrate the MeOH production process. Syngas—which often comes from an upstream gasification and/or steam reforming process—is enters the MeOH synthesis reactor section. A series of flash drums and stripping columns are then used to separate out any light ends, non-condensing gases and unreacted syngas, after which the crude methanol feed is sent to the distillation section for further purification. This crude methanol feed mainly contains MeOH, water, and DME, and it is in the first

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[4] At the elevated pressure of 10 bar, DME boils at approximately 40^{o}C. Cooling water (25^{o}C) is not adequate to cool and allow for condensation of the distillate.

distillation column—a topping column—where DME, is removed as distillate (top). The now enriched MeOH stream leaving out the bottom of the first column continues on to the second column, where waste water and, if present, other heavier alcohols (such as ethanol, propanol) are recovered in the bottoms (alternatively, heavier alcohols products maybe be drawn as side of the column (Ott et al., 2011),(Zhang, Liang, & Feng, 2010)), while high purity MeOH product is recovered as the distillate; however, here we are only focusing on the case where the waste water ends up in the bottoms, with no side draws permitted.

The MeOH product column, that is, the second column in the sequence, operating pressure can be at ambient conditions (A. a Kiss, Suszwalak, & Ignat, 2013) or above (Clausen et al., 2010),(Salkuyeh & Adams, 2014) depending on preference. In order to later have the conventional separation processes be comparable to the proposed intensified process, the column pressures in the MeOH section are taken to the same as in the DME section discussed earlier above.

3.2.2 Intensified Flexible Design Case: DME & MeOH Purification

The proposed design must, depending on process needs and/or market conditions, be able to switch between processing two different feeds: one feed being DME-rich and the other being MeOH-rich. In each case, chemical grade DME is collected from the distillate of the first column, while chemical grade MeOH is collected as distillate from the second column. The throughput for each product (DME or MeOH) varies as the feed is changed, but the product purity (wt. %) desired remains the same. If more DME is required at any given point, then the feed selected is that coming from the DME reactor since this allows for a high distillate flow rate. Conversely, if more methanol product is required, the feed is switched to that coming from the MeOH reactor to allow for increased distillate flow rate in the refining column. This way the process remains flexible in the face of changing market conditions. A schematic representing the envisioned process under study is shown in Figure 3.2.

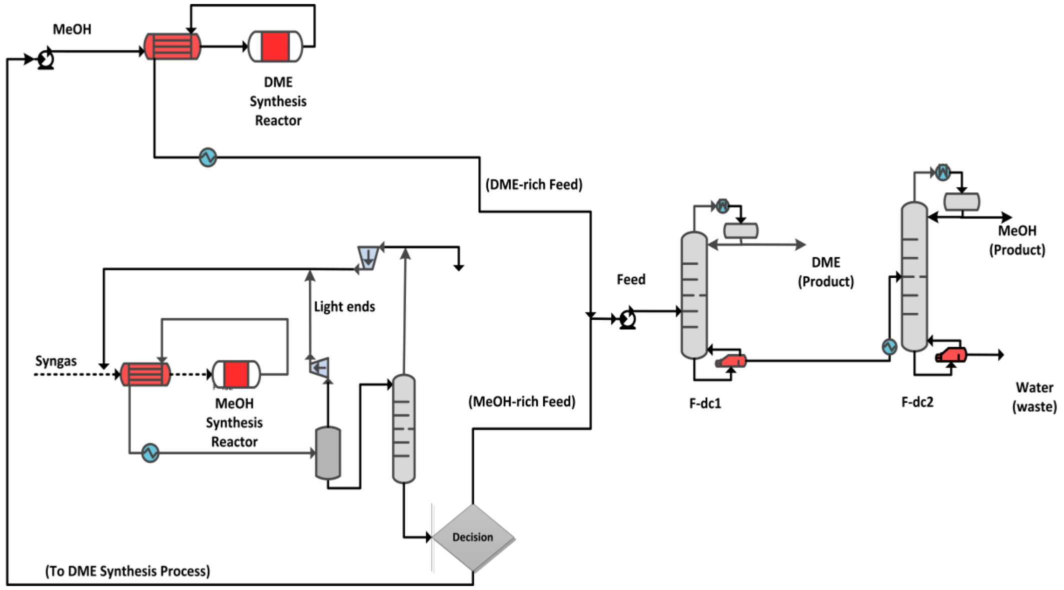

Figure 3.2. Intensified flexible distillation sequence for the separation of DME and MeOH. The proposed design accommodates switching of the feed stream between crude DME and crude MeOH.

The physical design parameters are fixed regardless of the feed being processed at any given point; however, the operating parameters can, via control architecture, be fine-tuned correspondingly with each feed. Whereas the height, diameter, and feed tray location of each column are fixed parameters, the reflux and boil-up ratios (and by extension, the heat exchangers duties delivered at the reboiler and condenser) allow for adjustment and fine-tuning during operation. The envisioned system has two distinct modes: the first—denoted as DME Operating Mode (DOM)—sees all of the crude MeOH go first to the DME synthesis reactor before finally being sent to the distillation section; while the second—denoted as MeOH Operating Mode (MOM)—has all of the crude MeOH from the MeOH synthesis reactor sent directly to the distillation section.

3.3 Simulation

The simulation of all process designs is carried out in the Aspen Plus (“Aspen Plus,” 2015) environment. The property package used to predict and characterize thermodynamic and transport characteristics of the vapour-liquid-equilibrium (VLE) of the components is UNIQ-RK. This is a property package that employs the Reidlich-Kwong (Redlich & Kwong, n.d.) equation of state to calculate the gas phase fugacity coefficient, coupled with the UNIQUAC (Abrams & Prausnitz, 1975) liquid activity coefficient model for determining the excess Gibbs free energy. The binary interaction parameter values used are available from within Aspen Plus. Prior work by other researchers validated the binary interaction parameters against experimentally reported values from the literature (A. A. Kiss & Ignat, 2013).

The first step is to get a rudimentary estimate of the reflux ratio and number of stages required for a particular separation; Aspen Plus’s shortcut column, DSTWU, is used. An approximate (shortcut) method, such as DSTWU, allows for estimates of the minimum number of equilibrium stages, feed tray location, and minimum reflux ratio to be established for a given separation. During the approximation, the Fenske method is used to determine the minimum number of stages (at total reflux), while Underwood's method is employed in determining the minimum reflux ratio. The rest of the values—for number of stages and corresponding reflux ratios—which lie between the minimum and total reflux limits established earlier by the Fenske and Underwood methods[5] are calculated using the classic Gilliland correlation (King, 1971).

After the approximate modeling step above, a more rigorous approach is chosen which makes use of Aspen Plus's rigorous distillation column model—RADFRAC. Output data from DSTWU is fed to RADFRAC and serves as an initial guess for the rigorous simulation. Murphree tray efficiencies are set at a constant 85%, a value that has previously been shown (Tock, Gassner, & Maréchal, 2010) to be a good approximation of the non-ideality (degree of non-equilibration at each stage) of the distillation tray model. All simulations assume steady-state column operation.

3.3.1 Column Modelling Details: MeOH Operating Mode

The base conditions for crude MeOH feed are assumed to be within the typical range of 70-90 mol% MeOH in crude methanol feed (Zhang et al., 2010). In order to aid comparison of the two separation processes during analysis in subsequent chapters of this work, a feed flow rate of 22,880 kg/hr—identical to the DME separation case—is adopted. In addition to the feed conditions, other critical process design parameters for the distillation columns are also listed in Table 3.1. The values displayed correspond to

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[5] Aspen Plus specifically makes use of Winn's correlation to correct for non-constant relative volatilities throughout the column.

the initial simulation case before preforming any optimization. The output of the simulation is used as a starting point for the optimization.

Table 3.1. MeOH operating mode: distillation process simulation details

Number of stages : DC1, DC2	45, 60
Feed stage: DC1, DC2	23, 27
Feed flow rate (kg/hr.)	22880
Feed molar composition (MeOH:DME:H_20)	0.82 : 0.15 : 0.05
Feed Temperature (K)	393
Column pressure (bar): DC1, DC2	10, 1
Tray type; spacing (m)	Sieve; 0.0062
DC1 Condenser utility	Refrigerant
DC1 Reboiler utility	MP Steam
DC2 condenser utility	Cooling Water
DC2 reboiler utility	MP Steam
MeOH distillate purity (wt. %)	99.85
Waste Water purity (wt. %)	> 99.9

3.3.2 Distillation Column Modelling Details: DME Operating Mode

The crude DME feed conditions is depended on the conditions of the effluent from the MeOH synthesis reactor. Using a conversion of 79% and a known reaction pathway in which methanol is catalytically converted to DME and water; the effluent conditions of the DME synthesis process are readily calculated. Pertinent simulation processes set up details, including the feed condition, are shown in Table 3.2. The distillation column (DC) design parameters listed therein are those used in the base case simulation, the results of which serve as a starting point of the optimization in the subsequent section (3.4).

Table 3.2. DME operating mode: distillation process simulation details

Number of stages : DC1, DC2	45, 60
Feed stage: DC1, DC2	23, 30
Feed flow rate (kg/hr.)	22,880
Feed molar composition (MeOH:DME:H_20)	0.17 : 0.34 : 0.49
Feed Temperature (K)	393
Column pressure (bar): DC1, DC2	10, 1
Tray type; spacing (m)	Sieve; 0.0062
DC1 Condenser utility	refrigerant
DC1 Reboiler utility	MP steam
DC2 condenser utility	Cooling Water
DC2 reboiler utility	MP steam
DME distillate purity (wt. %)	99.95

3.4 Optimization

3.4.1 Overview

The aim of the optimization is to design a flexible distillation process with the lowest total annualized cost (TAC) realizable, while meeting purity specifications on all component products (DME, MeOH, and waste H_2O). TAC takes into account the combined effects of annualized total direct cost (cost of installed equipment) and annual operating cost (cost of utilities) to yield a weighted value of the total cost of the process. A single optimal design with fixed physical parameters is being sought for the separation of two very different feeds; however during the optimization the operating variables will be kept free and adjustable, varying depending on the requirements of the feed being processed. Specifically, variables such as reflux and boil-up ratios and utilities flow rates to the condenser and reboiler can all be adjusted in accordance with the requirements of each operating mode, respectively for DM and MOM.

Recalling that we seek to determine principally the optimal number of stages and feed location for our proposed flexible design. An intuitive approach for evaluating—individually—the effect of the length of the rectifying and stripping sections on column performance involves tracking the effect of changing either the *number of rectifying stages* (number of stages above the feed location) and/or the *number of stripping stages* (number of stages below the feed). This is the approach adopted throughout the rest of this work.

3.4.2 Justification for the Chosen Approach

A closer look at the optimization problem as postulated—minimize total annualized cost of a distillation column while meeting purity specifications on both the distillate and bottoms streams—reveals that this comprises solving a series of continuous optimization problems nested inside a discrete optimization framework. However, it is worth noting that fixing the two integer variables (number of stages above feed and number of stages below feed) leaves no remaining independent variables[6]. It is well known that economic optimization—for optimal feed location and number of stages—for a distillation column gives rise to a mixed integer nonlinear programming (MINLP) problem, but instead of attempting to solve the problem using classical approaches (Swartz & Stewart, 1986), (Ricker & Grens, 1974), (Kister, 1992) or stochastic algorithms (Ramanathan et al., 2001), we exhaustively enumerate all possible integer variables combinations, and thereafter solve each instance of the continuous problem. As noted before, removing

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[6] Optimal reflux and boil-up ratios are still unknown, but these are not *independent* variables; for a given design with known number of stages and feed location, optimal reflux boil-up ratios exist (within given bounds).

integer variables eliminates degrees of freedom, which means the resulting problem that we now need to solve comprises simply a simultaneous system of nonlinear equations which can be readily solved using an appropriate technique such as, Newton's Method. Applying procedure for a (finite) combination of number of rectifying stages and stripping stages (the original integer variables) ensures that the entire optimization search space is explored. When all function values are available for the entire problem space, it becomes trivial determining the optimum (the value with the lowest cost).

3.4.3 Design Specifications and Sensitivity Analysis.

Each problem—comprising a system of simultaneous nonlinear equations (and inequalities) --mentioned above is solved in Aspen Plus utilizing the "design spec" and "vary" tools. The algorithm used is "Newton"—which is a method that implements a modified Newton's Method; in which derivative information is only calculated if the convergence rate is unsatisfactory, and a line search is also used to improve stability. In order to enforce the constraint for the light key component purity in the distillate for each of the two distillation columns, a "design spec" and "vary" tools are used in each case. In addition, a second "design spec" and "vary" pair on the light key impurity in the bottoms stream of (of each column) is invoked. Each "design spec"—essentially a greater-than-or-equal-equal-to constraint—paired with a corresponding variable (termed "vary" in Aspen Plus) that gets manipulated during the simulation in order to satisfy the given design specification. In this work, the light key purity in the distillate is controlled by adjusting the reflux ratio, whereas the light key impurity specification in the bottom stream is controlled by adjusting the boil-up ratio. After each simulation run is converged (using the design spec-vary function), a sensitivity analysis is performed on all combinations of the number of stages above and number of stages below feed.

3.4.4 Distillation Process Equipment Sizing

Equipment sizing is a crucial step in the design and, especially, the optimization of distillation columns. A tray-type column is the best choice for the separations being performed. The column uses sieve trays (arranged in a single pass flow fashion), employs a partial kettle reboiler, and also has a total condenser, all of which need to be sized appropriately. Major pieces of equipment whose dimensions are required include the condenser and reboiler (the surface area available for heat transfer), the column (the height and diameter of the column), column internals (the number of sieve trays[7]), and the sizes of the reflux drum and reflux pump. All other auxiliary pieces, such as valves and pipes, constitute a relatively small portion of the total column cost and therefore excluded from the equipment sizing and costing evaluations. When all of the dimensions for these major pieces of equipment have been successfully determined, the column performance can be accurately estimated.

All pertinent formulae for the calculation of various physical parameters of the distillation column are performed natively in Aspen Plus. Costing for all equipment is done offline, employing the methodology of Guthrie given in the work of Turton and colleagues (Turton, Bailie, Whiting, Shaeiwitz, & Bhattacharyya, 2012) (Rangaiah, Ooi, & Premkumar, 2009)

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[7] The number of trays is equal to the total number of stages calculated but excluding the condenser and reboiler.

3.4.4.1 Column diameter and height

The diameter of the column is automatically calculated in Aspen Plus, given all other inputs: number of stages, feed stage, feed flow conditions, column pressure, and distillate and bottoms purities. The velocity of the vapor stream within the column is the main determinant of column diameter, and in Aspen Plus, the Fair Correlation is implemented. The maximum vapor velocity allowable before entrainment and flooding occur is set as the upper limit, all allowable vapor velocities can at most be 85% of this value. Typically, a lower bound on vapor velocities is also enforced to prevent weeping, and this is usually about 50-60% of the maximum vapor velocity. The Fair Correlation essentially uses these bounds and other factors (including column tray spacing height, liquid viscosity, vapor and liquid densities, and pressure) to estimate the minimum diameter of the column whose design ensures no flooding occurs. There is usually no risk of weeping since the column is operated at close to the flooding limit.

For the column height calculation, two important factors are the total number of trays and the corresponding tray spacing between them. The column height is a function of total number of these two variables. In addition, the final column height will include an extra 15% of the calculated height; this is a heuristic that is taken from common industrial practice for real-world installed columns. The final column height calculated accounts for the extra space required for feed installation, and for easy service, maintenance, retrofitting of auxiliary equipment controls, and for various piping and plumbing requirements (such as, to deliver heating and cooling to and from the heat exchangers). Using variables available from within Aspen Plus simulation results the corresponding calculations necessary for determining the diameter and height are performed, also with Aspen Plus.

3.4.4.2 Condenser and Reboiler areas

Condenser and reboiler areas are directly related to column size (number of stages); as number of stages increase, the reflux ratio required to achieve same separation decreases. Consequently, condenser and reboiler duties decrease. Since heat exchanger area is proportional to heat duty, a decrease in heat duty results in comparatively less surface area required for heat transfer.

For a given distillation column of known diameter and known distillate and bottoms temperatures, the surface area needed for heat exchange in the condenser and reboiler can be determined. A log mean temperature difference (LMTD) –assuming counter-current flow in the heat exchangers—along with the heat duty delivered, the flow rates and temperatures of the streams being heated or cooled, and the overall heat transfer coefficient, are used in the classic heat balance equations for hot and cold streams in shell and tube heat exchangers to estimate the surface area required.

First, the surface area is obtained from a simulation run of value of the OHTC is derived from known values from the open literature (Edwards, 2008). An additional check of the OHTC value is performed using an iterative guess-and-check approach in which an initial OHTC is assumed and then that value is adjusted until it satisfies the given values of heat transfer rate, Q, the LMTD, and the heat exchanger surface area from calculated by

Aspen Plus. The final OHTC value used is kept constant throughout various scenario analyses while new surface area values are recalculated.

3.4.5 Distillation Process Costing

After successfully determining the sizes for all major pieces of equipment, the projected capital and operating cost of the process are subsequently estimated. Distillation column design optimization involves striking a balance between operating and capital costs, which have an inverse relationship[8]. Column diameter and number of stages both have the biggest impact of capital and operating costs. A trade-off exists between capital and operating cost. . Generally, the total cost—the sum of annualized capital cost and operating cost--of distillation is highest at minimum reflux (the so-called “infinite number of stages” scenario) and again at total reflux (when the stage requirements are minimum). In the region bounded by these theoretical limits, the individual weight contributions of the operating cost and the capital cost are variable; such that, for certain ranges of the reflux ratio, the total cost goes down as the reflux ratio is reduced, while in other ranges of the reflux ratio the opposite behavior is observed. This is because operating cost (mainly energy utilization costs) increase monotonically with increasing reflux, whereas the effect of reflux on capital cost is not always monotonic. Reflux affects flow rates within the column, thus having an impact on the column diameter. At low reflux ratios this impact is minimal when compared to the weight of operating cost; however, at high reflux ratios the impact of the increased capital cost (due to increased diameter) begins to

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[8] Although this inverse relationship is valid only for certain regimes of the design optimization space; there comes a point in the design process when both capital and operating costs move in the same direction.

outweigh that of the operating cost. There therefore exists a region when these two costs' combined weight leads to the lowest total cost. Design optimization approaches attempt to exploit this phenomenon.

In our optimization approach, scenarios corresponding to all combinations of number of stages and feed location are individually sized and costs subsequently estimated and ranked—the design with the lowest cost (TAC) is favored.

3.4.5.1 Capital Cost

The capital cost for each process comprises the column shell cost, cost of sieve trays, and reboiler and condenser costs. To estimate the total direct cost an installed cost factor (obtained from base case simulations, using Aspen Capital Cost estimator tool in Aspen Plus) of 2.96 is used. This is the ratio of the installed cost to the purchased cost of the base case design. This value is used in subsequent scenarios analyses, due to Aspen Capital Cost estimator being unavailable during sensitivity runs. Total direct cost accounts for the purchase cost of the equipment, installation (including piping, instrumentation and labor), taxes, supervision, and shipping cost (Biegler, Grossmann, & Westerberg, 1997).

3.4.5.2 Operating cost

In distillation processes, energy costs constitute the bulk of the operating costs. In this work, the operating cost considered includes only the energy cost of the reboiler (cost of hot utility) and the condenser (cost of cold utility). The hot utility is medium pressure (MP) steam, while the cold utilities are refrigerant and cold water, respectively for DME and MeOH separation. Utility costs are influenced largely by the reflux ratio and to a lesser extent by the boil-up ratio. The reflux ratio affects the vapour flow rate within the column, which in turn dictates the column (diameter) and condenser sizes. An increased vapour flow rate in the column requires a larger heat exchanger surface area and/or higher utility flow rate in the heat exchangers to meet the required heat duty. A higher rate of utility usage leads to higher operating cost. The total annual operating cost is computed from the product of hourly utilities cost and using a service factor of 0.956 (or 8400 hours of operation per year).

3.4.5.3 Total Annualized Cost

An interest rate of 10% and a lifetime of 15 years are assumed in calculating the annuity factor, used in determining annualized total direct cost. The sum of the annual operating cost and the annualized total direct yields the total annualized cost (TAC). The following equations illustrate this approach:

$$A_f = \frac{i(1+i)^t}{(1+i)^t - 1} \tag{1}$$

$$TAC = A_f * Total\ Direct\ Cost + Operating\ Cost \tag{2}$$

In equation (1), “A_f” is the annuity factor, “i” is the fractional interest rate per period, and “t” is the number of periods (years). The above methodology is adapted from the work of Smith (Smith, 2005, p. 24).

3.4.5.4 Algorithm for determining minimum TAC

An algorithm is devolved for the realization of the best overall design—one which meets all product purity requirements, regardless of the feed being processed. Such an optimal design necessarily has the lowest TAC amongst all other alternatives. Both components of TAC—annualized total direct cost and annual utility cost—are individually evaluated. The optimization of TAC is performed for each *pair* of feed-column combination. An optimal column design—one with minimum TAC—is determined for the following combinations: DME operating mode /Column 1 and MeOH operating mode/column 1, and for DOM/Column 2 and MOM/Column 2. The sum of the two TAC values for columns 1for both operating modes and column 2 (also for both modes) is the TAC for the overall intensified flexible distillation design. The minimum TAC is a function of the fraction of time spend in each operating mode.

Table 3.3. Four different feed-column combinations for flexible distillation design

Feed Type	Distillation Column Sequence #
DME-rich feed	1
DME-rich feed	2
MeOH-rich feed	1
MeOH-rich feed	2

A fundamental task of the algorithm is to address the critical step in the problem formulation that involves determining the *fraction of time in a given operating year* each feed is expected to be processed. The fraction of time is determined for increments of 1%, from 0% to 100% for each mode. For a given operating time for each feed, the annual utility cost corresponding to each feed is readily determined. Within the algorithm, the other component of TAC—annualized TDC—is determined, following first equipment sizing and then costing of the selected sizes. The equipment sizes are chosen from the greatest common multiple for each physical parameter (column diameter, heat exchanger areas) between values for the two feeds.

It is crucial to note that the algorithm for estimating the design with the lowest TAC operates with Aspen Plus simulation data, using each sensitivity analysis scenario's results, but performed entirely offline. Implementation of the algorithm is carried out in MATLAB ("MATLAB," 2014). During a major iteration (corresponding to selecting a column with a specified number of stages above and below the feed) the following parameters are calculated:

- Equipment sizes
    - Optimal column diameter: taken as the largest diameter between the column diameter for DOM and MOM, rounded up to the nearest 0.5 ft.
    - Optimal heat exchanger areas: taken as the maximum of DOM and MOM heat exchanger areas.
- Capital and Operating Costs
    - Optimal heat exchanger cost: taken as the sum of the cost of the condenser with the calculated optimal surface area, and the corresponding cost of the reboiler of optimal surface area
    - Optimal column cost: taken as the cost of the column with the optimal diameter calculated.

  - Optimal utility cost: taken as the sum of the cost of cold utility for the calculated optimal condenser area, and the cost of hot utility for the calculated optimal reboiler area
  - Optimal Total direct cost: taken as the sum of the following costs: optimal column cost, optimal heat exchanger cost, all escalated by an installation factor.
- Total annualized cost
  - Taking into account the number of hours of operation of the column with DME-rich feed connected, and the number of hours of operation of the column with MeOH-rich feed connected, the minimum value among all combinations of TAC is calculated

Mathematical formulation

Intensified Flexible Design

The algorithm for calculating the minimum TAC for the proposed design can be approximated by the following mathematical statements:

$$TAC_{sytem} = \sum TAC_i \tag{3}$$

$$i = \text{f-dc1}, \text{f-dc2}$$

The TAC_i are found by solving the following optimization problem:

$$\underset{N_{A,i},N_{B,i}}{\text{Minimize}}\ Total\ Annualized\ Cost_i \tag{4}$$

subject to:

$$h_j\left(x_j,\ N_{Ai},\ N_{Bi}\right) = 0, \qquad j = \text{DOM, MOM} \tag{5}$$

$$x_j = \{\mathrm{RR_{i,j}}\,, \mathrm{BR_{i,j}}\,, \mathrm{Q_{C_{i,j}}}, \mathrm{Q_{R_{i,j}}}\,, \mathrm{A_{C_{i,j}}}, \mathrm{A_{R_{i,j}}}, \mathrm{D_{i,j}}\,, \dots\}$$

$$A_{C_i} = \max\ [A_{Ci,DOM}\,, A_{Ci,MOM}\,] \tag{6}$$

$$A_{R_i} = \max\ [A_{R_i,DOM}\,, A_{R_i,MOM}] \tag{7}$$

$$D_i = \max\ [D_{i,DOM}\,, D_{i,MOM}] \tag{8}$$

$$TDC_i = f1\left(A_{C_i}\right) + f2\left(A_{R_i}\right) + f3(N_{A_i}, N_{B_i}, D_i) \tag{9}$$

$$\begin{aligned} Operating\ Cost_i &\\ &= \phi\left[Cost_{QC} * Q_{C_{i,DOM}} + Cost_{QR} * Q_{R_{i,DOM}}\right] \qquad (10)\\ &+ (1-\phi)[Cost_{QC} * Q_{C_{i,MOM}} + Cost_{QR} * Q_{R_{i,MOM}}] \end{aligned}$$

$$TAC_i = A_f * TDC_i + Operating\ Cost_i \tag{11}$$

In equations 3 to 11 above, $h_j(x_j, N_{A_i}\,, N_{B_i}\,) = 0$ represents all model equations for the distillation column (that is, the `RADFRAC` model in Aspen Plus), including the “design spec” for product purities. Each distillation column is indexed by i, while j denotes each of the two operating modes, based on Figure 3.2. N_{A_i} , N_{B_i} are the decision variables of the optimization, while x_j are the continuous variables. All other abbreviations are as previously defined.

Conventional Design

For the conventional design, the mathematical formulation is slightly different; however, the essential parts of the framework employed in the intensification approach persist. The relevant equations are provided below:

$$TAC_{sytem} = \sum TAC_i \tag{12}$$

$$i = \text{cDME-dc1, cDME-dc2, cMEOH-dc1, cMEOH-dc2}$$

$$\underset{N_{A,i}, N_{B,i}}{\text{Minimize}}\ Total\ Annualized\ Cost_i \tag{13}$$

$$\text{subject to:}$$

$$h(x,\ N_{Ai},\ N_{Bi}) = 0,$$

$$x = \{RR_i, BR_{i,j}, \mathrm{Q}_{\mathrm{C}_i}, Q_{R_i}, A_{C_i}, A_{R_i}, D_i\} \tag{14}$$

$$Total\ Direct\ Cost_i = f1\left(A_{C_i}\right) + f2\left(A_{R_i}\right) + f3(N_{A_i}, N_{B_i}, D_i) \tag{15}$$

$$\begin{aligned} Operating\ Cost_i &= \phi\left[Cost_{QC} * Q_{C_i} + Cost_{QR} * Q_{R_i}\right]\delta_i + (1-\phi)[Cost_{QC} \\ &* Q_{C_i} + Cost_{QR} * Q_{R_i}](1-\delta_i) \end{aligned} \tag{16}$$

$$\delta_i = \begin{Bmatrix} 1 & \text{for } i = \text{cDME-dc1, cDME-dc2} \\ 0 & \text{for } i = \text{cMEOH-dc1, cMEOH-dc2} \end{Bmatrix} \tag{17}$$

$$TAC_i = A_f * TDC_i + Operating\ Cost_i \tag{18}$$

3.5 Summary

In this section, a novel process intensification scheme—the design of an intensified flexible distillation process for the separation of DME and MeOH—is presented. This unique, innovative approach is aimed at exploiting the hidden benefits (cost savings) that can be reaped from reducing a plant’s installed equipment and overall footprint. The proposed trimming of installed equipment is realized by essentially repurposing all of the distillation equipment used in one separation process (DME) for use in another (MeOH). In order to realize such a flexible design, capable of meeting separation requirements for two distinct processes, design optimization is undertaken. Optimization takes into account the anticipated distinct operating modes for the process. Necessarily, the design must be optimal for a so-called MeOH operating mode (MOM), while at the same time being optimal for the DME operating mode (MOM), on a total annualized cost (TAC) basis. The minimum TAC will depend entirely on the fraction of time spent in each of the two operating modes or, equivalently, on the length of each period of operation.

Chapter 4 INTENSIFIED FLEXIBLE PROCESS VS CONVENTIONAL PROCESS

4.1 Introduction

The previous chapter provided an exposition of the process modelling and economic optimization of the two—conventional and intensified flexible—designs for the separation of DME and MeOH. The methodology involves the minimization of cost (TAC), the solution of which yields the desired optimal design. In this chapter, the results of the modelling and optimization are presented.

First, the lower and upper bounds on critical column design variables, namely the number of stages and internal ratios (reflux and boil-up), are established. Table 4.1 shows the bounds selected. Results obtained from the DSTWU shortcut method yield the lower bounds on N_S and RR, whereas the upper bounds on those variables are determined via process knowledge about the design of distillation columns of each of DME and MeOH separation.. From this bounding process, it becomes clear that optimal designs for each process lie somewhere within the range demarcated by the bounds. The lower bound sets the absolute minimum column stages required to achieve the separations desired, with the minim reflux ratio bound also included. Since a true upper bound on the size of the column or the magnitude of the reflux ratio does not exist, upper bounds are chosen based on process knowledge and practical limits.

Table 4.1 Upper and lower bound values on the number of stages and reflux ratio for columns used in the conventional DME-MeOH separation design

	cDME-dc1	*cDME-dc2*	*cMEOH-dc1*	*cMEOH-dc2*
$\mathbf{N_{S\_min}}$	12	16	10	18
$\mathbf{N_{S\_max}}$	45	60	45	60
$\mathbf{RR_{min}}$	0.935	1.029	11.734	0.397
$\mathbf{RR_{max}}$	40	40	100	40
xD	≥ 0.9995	≥ 0.997	≥ 0.999	≥ 0.9985

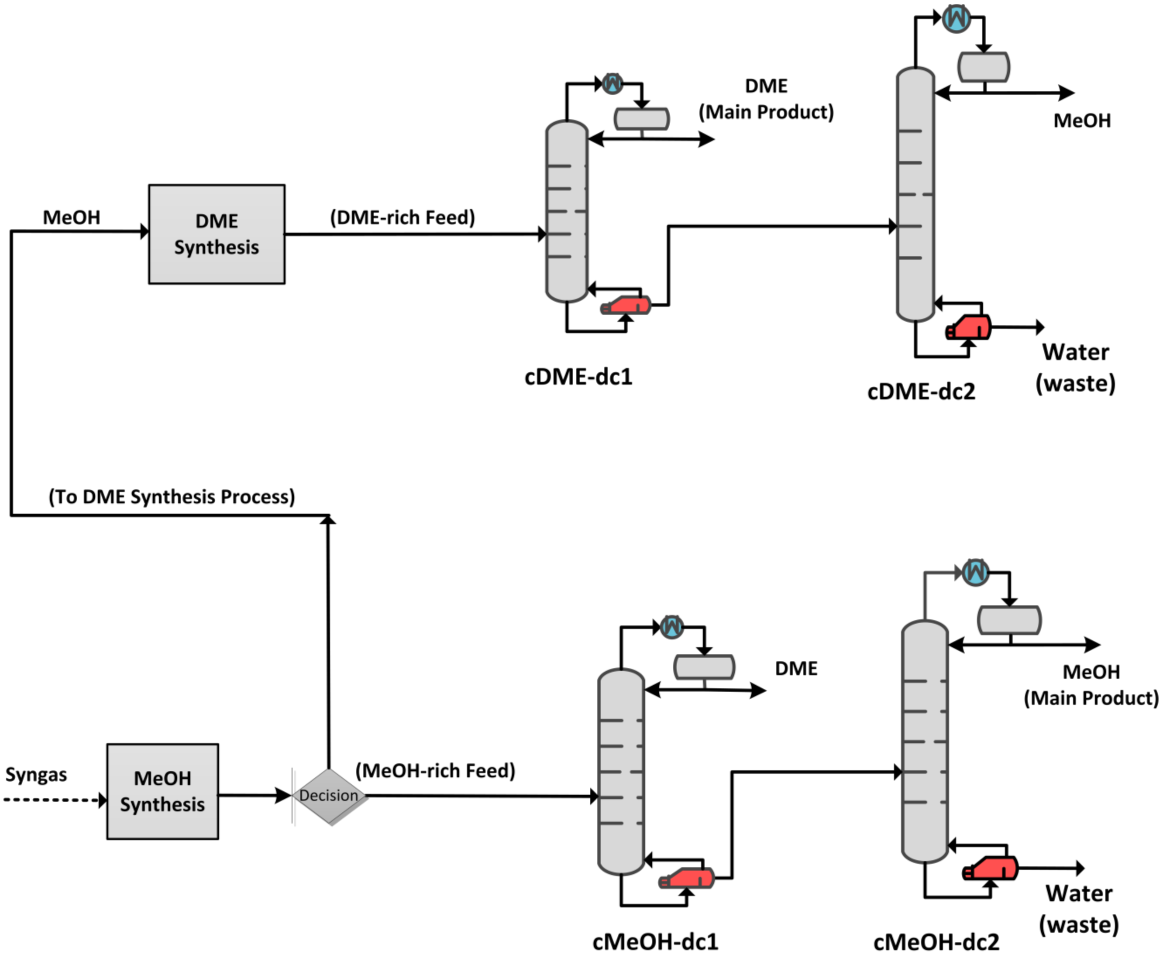
A process flow diagram of both the conventional and flexible designs, with simplifications made to the upstream synthesis processes, is shown in **Figure 4.1**. The simplification to the reaction processes is made in order to highlight the distillation sections, the examination of which is the focus of this work. Importantly, since the reactor sections remain fixed between designs, these are ignored throughout (**Figure 3.1** and **Figure 3.2** for the detailed processes showing full reaction sections can be consulted). In each process flow diagram, the blue lines delineate the design in which the plant is in DME Operating Mode (DOM), while the default (black) sections represent MeOH Operating Mode (MOM). A decision on which mode is activated is made at the methanol reactor outlet; either the reactor product is upgraded to yield pure MeOH product (for sale) or it is directed to the DME synthesis section of the plant where pure DME is removed as product. It should be noted that both DME and MeOH products are made in both modes, the main difference is in the throughput values; in DOM the majority of product is DME while in MOM the main product is MeOH. In addition, the length of time that the plant is kept in any of the two operating modes is the principal variable of interest in the optimization.

List of labels used

Label	Description
DOM	DME Operating Mode[1]
MOM	MeOH operating mode[2]
cDME	Conventional Distillation Design Process, DME operating Mode
cMeOH	Conventional Distillation Design Process MeOH operating mode
fDME	Flexible Distillation Design Process, DME operating mode
fMeOH	Flexible Distillation Design MeOH operating mode
cDME-dc1	Conventional Distillation Design, DME operating Mode, Column #1
cDME-dc2	Conventional Distillation Design, DME operating Mode, Column #2
cMEOH-dc1	Conventional Distillation Design, MeOH operating Mode, Column #1
cMEOH-dc2	Conventional Distillation Design, MeOH operating Mode, Column #2
c-dc1&2	Conventional Distillation Design, Column #1 & Colum #2
c-dc1&2	Conventional Distillation Design, Column #1 & Colum #2
f-dc1	Flexible Distillation Design, Column #1
f-dc2	Flexible Distillation Design, Column #2
φ	Fraction of time spent in DME operating mode

[1] DME reactor product as feed
[2] MeOH reactor product as feed

***(a)** Conventional Design*



***(b)** Intensified Flexible Design*

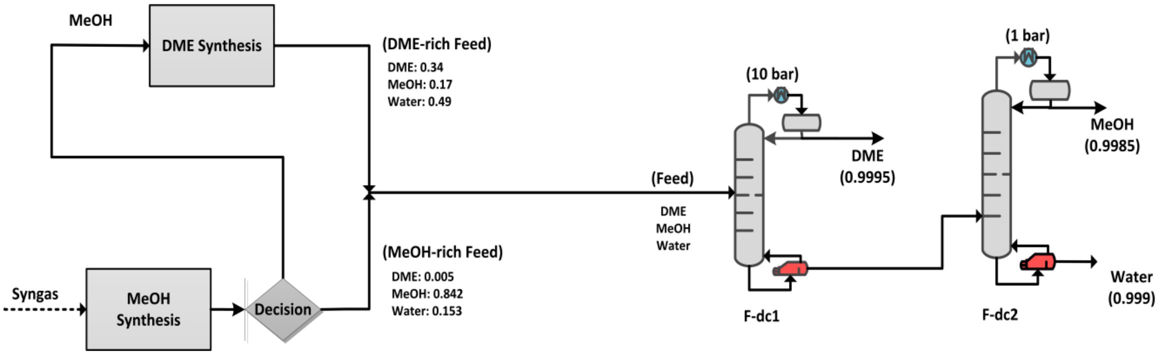


Figure 4.1 Conventional and Flexible Design Processes for DME-MeOH Separation

4.2 Simulation

Table 4.2 Utility costs

Utility	$/GJ
HPS	$ 2.50
MPS	$ 2.20
LPS	$ 1.90
CW	$ 0.21
REFRIG	$ 3.36

The modelling results of the conventional DME-MeOH separation process of **Figure 4.1** are provided. **Table 4.2** provides the cost of utilities used in all simulations**.** Table 4.3shows the bounds on the number of stages and reflux ratio for all four columns of the conventional process, and for each of the two operating modes. Corresponding columns in each operating mode have the same maximum number of stages ($N_{S\text{-}max}$) in both modes; that is, cDME-dc1 (column 1 in *DME Operating Mode*) and cMEOH-dc1 (column 1 in *MeOH Operating Mode*) each have at most 45 stages, while cDME-dc2 and cMEOH-dc2 (the second columns in each operating mode) each have at most 60 stages. However each column has different minimum number of stages (N_{S_min}) as these are uniquely determined by the Fenske-Underwood-Gillard correlations discussed in Chapter 3 of this work.

The upper limit on the Reflux Ratio (RR_{max}) is set at 40 for all but the cMEOH-dc1 column, whose RR_{max} is set at 100. This higher value is required due to extremely low vapour flowrate into the condenser of the cMEOH-dc1 column (due to the low initial concentration of DME in the feed). Also shown in the table are constraints on purities of light key components in the distillate (xD) for each column. The light key component is

DME in all dc1 columns, while it is MeOH both dc2 columns. It is worth noting that xD values for cDME-1 and cMEOH-dc2 are the purities of DME product and MeOH product, respectively. The distillate purities on the other two columns are a set a little lower since these correspond to recycle streams.

Table 4.3 Feed conditions for each operating mode

Feed	***DOM***	***MOM***
F (kg/hr)	22880	22880
T ($^{\circ}$C)	115	115
P (bar)	10	10
q_F	0	0
x_{DME} (mol %)	0.34	0.005
x_{MeOH} (mol %)	0.17	0.842
x_{Water} (mol %)	0.49	0.153

4.3 Optimization Results

In the previous section on simulation results, only product purities constraints were enforced; that is, the main goal was establishing *feasibility* of the designs being sought. In this section, not only do the designs have to be feasible but they must also be of minimum cost (TAC); that is, *optimality* of the feasible designs is the main goal.

The optimization results differ from the simulation results discussed previously due to this key fact: for all analyses performed in this section, the total annualized cost (TAC) is minimized for all viable designs, for both conventional and flexible process designs. Section 4.3.1 provides a detailed explanation of how the optimization results are obtained.

Results from the conventional design process are independently discussed in the first subsection (4.3.2), followed immediately by an independent look at the intensified flexible design in the subsequent subsection (4.3.3). A comparative analysis of the conventional and flexible designs is presented in the third and final subsection (4.3.4) of this optimization results section.

4.3.1 Determination of Optimal Design based on TAC

The results of each simulation run for each distillation column, for all chosen scenarios (using Aspen Plus's Sensitivity Analysis tool on the number of stages above feed and the number of stages below feed), and for all values of phi—the fractional amount of time spent in DME operating mode—are presented. From the simulation, values for the column physical attributes are obtained and these are used in turn to calculate column capital costs; noting that condenser and reboiler duties—also obtained from the simulations—are used in determining operating costs.

Table 4.4 includes a list of the design variables for the optimization problem. The discrete optimization performed relies on manipulating these three sets of data: namely, design variables, phi, and the number of stages above and below the feed, to obtain the Total Annualized Cost for each column. The minimum TAC for each design is a minimized sum of all the TAC values for the individual columns.

Table 4.4 Variables of Interest for Distillation Column Design and TAC Determination

Design Variable
Diameter
Condenser Area
Reboiler Area
Condenser Cost
Reboiler Cost
Column Cost
Cost of Cold Utility
Cost of Hot Utility
Total Operating Cost
Total Direct Cost

Conventional Design

ϕ = 0 or phi =1 case:

When $\phi = 1$ the plant is always in DME operating mode (DOM), wherein DME-rich feed from the process is being purified as showed the *top* section of Figure 3.1. The $\phi = 0$ case corresponds to the case where only MeOH-rich feed gets processed by the separation train, depicted by the process in the *bottom* section of Figure 3.1. All designs, regardless of operating mode, will have only two distillation columns (since only one of the conventional processes physically exists at these limits). TAC determination for these cases comprises the following elements:

i. Determine annualized capital cost + operating cost of first column and second column, for either of DOM (ϕ=1) or MOM(ϕ=0). This corresponds to the TAC of each column in any one operating mode.
ii. Calculate the total TAC by summing up the two TAC values from (i).
iii. Determine the minimum TAC for the whole design by finding the lowest value of the sum of the TACs for the two columns in (ii).
iv. At the calculated minimum TAC, extract all other variables of interest—all of which TAC is a function—for each distillation column: column diameter, condenser area, reboiler area, total number of stages above feed, total number of stages below feed, reflux ratio, boil-up ratio, capital cost, operating cost.

0 < ϕ <1 case:

All designs comprise all four distillation columns of Figure 3.1, representing the case where both the MeOH-rich feed and DME-rich feed are being handled to some degree by the separation process. All operating costs for DOM are weighted using phi while (1- ϕ i) is the weight of operating costs corresponding to MOM. In a nearly similar manner to the case of extreme ϕ values handled above, TAC determination for this section comprises the following elements:

i. Determine Annualized capital cost + operating cost of first column and second column in both DOM and MOM. This corresponds to the TAC of each of the four columns.
ii. Calculate the total TAC by summing up the four TAC values

iii. Determine the minimum TAC for the whole design by finding the lowest value of the sum of the TACs for the four columns in part (ii)

iv. At the calculated minimum TAC, extract all other variables of interest—all of which TAC is a function—for each distillation column: D, A_c, A_r, N_A, N_B, RR, BR, TDC, UTL.

Intensified Flexible Design

The intensified design always comprises two columns and only one separation train as shown in Figure 3.2. TAC determination for in this case comprises the following elements:

$\phi = 0$ or $\phi = 1$ case:

TAC determination for the case when phi=1 and phi=0 is trivially determined since these two cases are identical to the conventional design case (at the same phi values).

$0 < \phi < 1$ case:

For this case, a compromise column is required , represented by f-dc1 and f-dc2 in Figure 3.2. The determination is done for one column at the time, with the overall TAC for the design taken as the sum of the TAC values for both columns. The following steps outline the procedure for determining the compromise column (this is done for each of f-dc1 and f-dc2):

i. First choose one operating mode, e.g. DOM, and then simulate the column for all combinations of number of stages above and below feed. Phi is kept at 1 in this initial step.
    - Determine all variables of interest as shown in Table 4.4, along with TAC. Store the results,; where each scenario row contains values of all the variables of interest
    - Next, iteratively recalculate operating costs for all 0 < ϕ <1, by increments of 1%. This means that both DOM and MOM are active, to varying degrees.
    - Calculate TAC at each NA, NB, and ϕ.

ii. Next choose the other operating mode, in this case, MOM, and then simulate the column for all combinations of number of stages above and below feed. ϕ is kept at 0 in this initial step to indicate pure MOM as an initialization point.
    - Determine all variables of interest shown in Table 4.4.+ TAC and save.
    - Iteratively recalculate operating costs for all 0 < ϕ <1, by increments of 1%
    - Calculate TAC at each NA, NB, and phi.

iii. Choose the minimum values of $TAC = f(NA, NB, \phi)$ between the two values determined in (i) and (ii) .

iv. At the calculated minimum TAC, recalculate the physical parameters (diameter, condenser area, reboiler area) by taking the maximum values of each for the two operating modes.

- Use the recalculated (compromise) values of the physical parameters to recalculate all other costs (condenser cost, reboiler cost, distillation column cost, capital cost)

The total TAC for the intensified flexible design is the sum of the TAC values of the two distillation columns in the separation train, f-dc1 and f-dc2.

4.3.2 Conventional Design

4.3.2.1 Conventional Stand-Alone Designs

Simulation results suggest there is ample room for improvement on the design of distillation column for the separation of DME and MeOH in the conventional process. Accordingly, results for optimal designs—those with the lowest TAC --are shown in **Table 4.5**. N_A, N_B, N_S, and N_F are the number of stages above the feed, the number of stages below the feed, the total number of stages, and the location of the feed stage, respectively. D is the column diameter (to the nearest half foot), RR and BR are the reflux and boil-up ratios, while TAC is the calculated lowest total annualized cost for each distillation column. The total TAC values of each process during each of the two operating modes (DME and MeOH) are also provided in the table. In Each process, DME is enriched in the first column while MeOH gets enriched in the second column.

There is a caveat to the results of **Table 4.3** and **figure 4.3** above : these results are for individually optimized—stand-alone--cases, wherein only one feed (either MeOH reactor effluent or DME synthesis reactor effluent) is separated into product DME and product

MeOH., Orr equivalently: the case when only one operating mode is active. Nevertheless these findings provide “early” impressions on the nature of the optimization problem when taking into account the possibility of making both DME and MeOH. The results represent extreme cases when the fraction of time spent operating in one mode is *fixed* and is exactly equal to 1.

Table 4.5 Summary of optimal column design parameters for the conventional DME-MeOH separation process. Results their TAC values are shown for stand-alone processes.

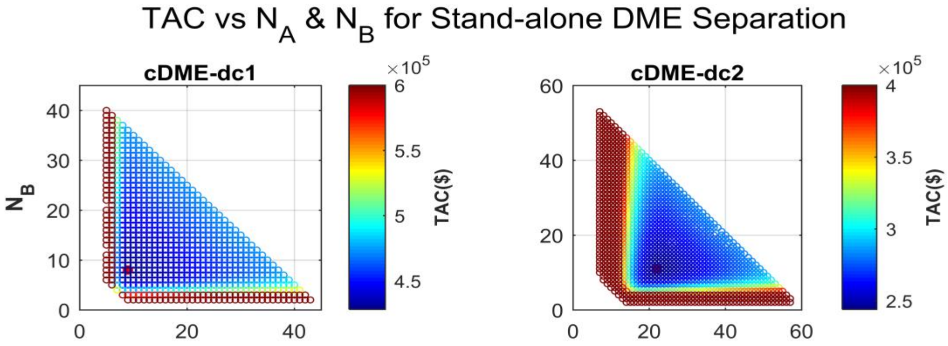
	N_A	N_B	N_S	N_F	D	RR	BR	TAC ($/yr)
cDME-dc1	9	8	17	10	4	1.706	0.184	430,000
cDME-dc2	22	11	33	23	4.5	1.075	0.826	240,000
DME-mode Total								**670,000**
cMEOH-dc1	9	18	27	10	2.5	22.4	0.170	180,000
cMEOH-dc2	40	9	49	41	8	0.807	7.263	860,000
MeOH-mode Total								**1,040,000**

It is clear that, on a cost basis, it is cheaper to *separate* the DME reactor product than it is to separate the MeOH reactor product, for the stand-alone processes. Furthermore, the following additional findings are made:

i. For the DME process, the more expensive column is the first (cDME-dc1), while the second column in the MeOH process (cMEOH-dc2) is responsible for the bulk of the cost (TAC). These high cost columns correspond to the sites where product DME and product MeOH are obtained.
ii. For both process modes, the columns in which MeOH is being enriched have the highest total stages requirement (33 and 49, respectively for cDME-**dc2** and cMEOH-**dc2**); whereas the columns in which DME is enriched have fewer stage requirements (17 and 27, respectively for cDME-**dc1** and cMEOH-**dc1**). Moreover, columns in which MeOH is enriched have larger rectifying sections compared to the columns in which DME is being enriched.
iii. In addition, columns in which MeOH is enriched have correspondingly larger diameters (4.5ft and 8ft, respectively for cDME-**dc2** and cMEOH-**dc2**) compared to the DME columns (4ft and 2.5ft, respectively for cDME-**dc1** and cMEOH-**dc1**).
iv. Therefore, MeOH separation requires tall, thick columns, in contrast to DME separation which requires relatively thin, shot columns overall, wherein cost minimization is the objective.

The behaviour of the total annualized cost during both operating modes as column number of stages is varied is further explored; the results are shown in **Figure 4.2**. The figure shows plots of colour maps for the two columns in each of the operating modes, the top row represents DOM while the bottom row shows the MOM results. The red dots in each chart are the points of minimum TAC (corresponding to the values given in **Table 4.5** above). Each discrete point plotted represents a feasible column design.

(a)



(b)

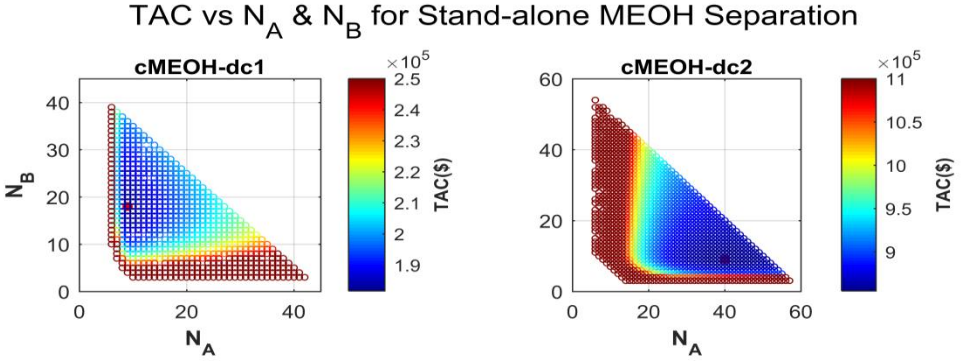


Figure 4.2 Total Annualized cost (TAC) vs no. of stages above feed (N_A) and no. of stages below feed (N_B) for stand-alone processes for DME & MeOH separation

The following observations are made from the plot results:

- cDME-dc1: the optimal column is the one with nearly equal rectifying (N_A) and stripping (N_B) sections. In addition, the fewer the overall number of stages (the shorter the column) the lower the TAC.
- cDME-dc2: a column with a large rectifying section (N_A) is more favorable, as shown by the pattern of the color scheme. This means the feed stage is located near the bottom of the column.
- cMEOH-dc1: a column with a large stripping section (N_B) is one that minimizes TAC
- cMEOH-dc2: a column with a large rectifying section (N_A) is more favorable. The feed stage is located near the bottom of the column. Overall, a relatively tall column is required for minimum cost.

4.3.2.2 Conventional design of DME and MeOH distillation

The analysis in this subsection will explore the effect of variable ϕ --defined as the fraction of time in an operating year dedicated to operating in the DME Operating Mode[9] (the top process depicted in part (a) of **Figure 4.1**). In the conventional design case, it is important to recall that there are two separate distillation trains, each comprising two columns, such that there are four columns in total. The exception is when only one product is being made at all times, in which case only one distillation train exists (or two columns in total)—this happens when ϕ is either zero or one. The interplay between the production of both DME and MeOH (for various lengths of time in a given operating year) is assessed. When accounting for the effect of ϕ, what was once an optimal design

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[9] Correspondingly, the fraction of time operating in MeOH mode is represented by $(1-\phi)$.

for each product is no longer optimal for the new design, thereby requiring complete redesign in order to achieve minimum cost under the new additional constraint imposed by ϕ. Since $TAC = f\ (NT, D, Qc, QR)$, and $Ac = f\ (\ Qc\ )$ and $A_R = f\ (\ Q_R\ )$, the physical design parameters of the distillation column (diameter, D, no. of stages, N_S area of reboiler, A_C, and area of condenser, A_R) need to be re-optimized for each different value of ϕ—which affects Q_C an Q_R. **Figure 4.3** shows the effect of varying ϕ (between 0 and 1) on the values of the physical design parameters which yield a column design of minimum cost (TAC). The figure shows all four columns—the two columns of DME Operating Mode (DOM) together with the two columns of MeOH Operating Mode (MOM).

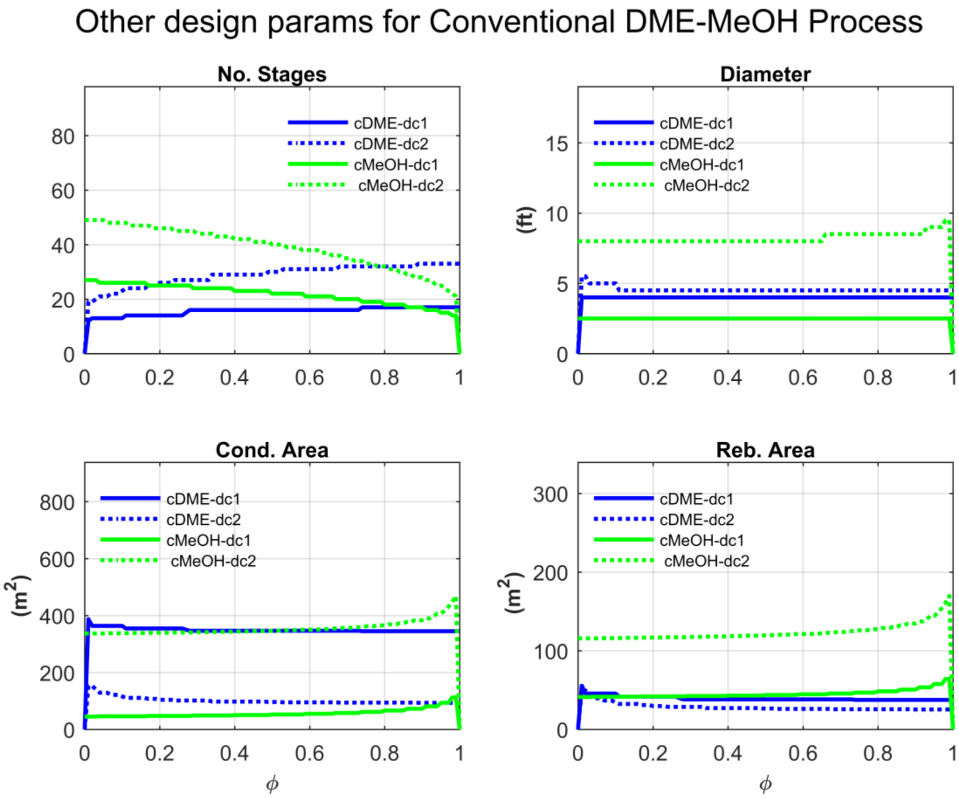

Figure 4.3 Optimal physical design parameters for the conventional design process. Values for Ns, D, A_C, and A_R versus ϕ for all columns are given for both DME (blue) and MeOH (green) operating modes.

When$\phi = 0$, the design represents the case of operating in MOM all year round; at the other extreme, when$\phi = 1$, the design corresponds to the case where only the DOM is active at all times. Both of these trivial cases were handled previously (the results of which appear in **Table 4.5**). Thus, the region of interest lies in the range $0 < \phi < 1$ and all future analysis will focus on this region. The apparent discontinuities (or “jumps”) observed for value of ϕ just after $\phi = 0$ and just before $\phi = 1$ are due to the fact that the endpoint values correspond to designs that are decidedly different from the ones in the delimited range.. As previously mentioned, at the end points only one product—either DME or MeOH—is made. Conversely, varying amounts of DME and MeOH product are made in the $0 < \phi < 1$ range. That is, there are only two distillation columns at the end points (when only one operating mode is active) compared to any other value of ϕ, where there are four columns. This means that all costs—capital and operating costs—are, correspondingly, that much lower at the end points compared to anywhere else in between the end points. The following further observations are made about the effect of ϕ on the optimal values of the four physical design parameters shown (in **Figure 4.3**):

- **No. of Stages**: Optimal N_S increases for both columns of the DOM (blue) as ϕ increases since increasingly more time being spent in DOM requires correspondingly larger columns. Increasing ϕ has the effect of increasing weight of operating cost component of TAC; therefore, in order to achieve minimum TAC, it becomes preferable to have a larger number of stages and correspondingly lower reflux ratio—which lessens the impact of the operating cost—in order to achieve the same level of separation desired at minimum total cost. The slight increase in capital cost—due to the higher number of stages--is tolerable when weighted against the alternative of having a comparatively larger (more heavily weighted) operating cost. In addition, higher reflux ratio would require a larger diameter column, leading to further cost increases (due to the increased capital cost of a large diameter column).

When in MOM, the opposite behaviour is observed for columns of the MOM (green); this is due to the fact that less MeOH product is made as the value of (1-ϕ) approaches zero. Less amount of product made means lower operating cost which means the weight of the operating cost component in TAC is reduced, while the weight of the number of stages component of capital cost is increased. Therefore, in order to minimize TAC, few stages are required as this has the most effect on TAC in this operating regime (as was discussed in 3.4.5 on distillation process costing).

- **Diameter**: as ϕ increases optimal D gets smaller for columns of the DOM, although D remains constant for the cDME-dc1 column. On the other hand, in MOM, D increases for cMEOH-dc2 but remains constant for cMeOH-dc1. This indicates that the value of ϕ has a bigger effect on the MeOH column (the second column of each mode) than on the DME column (the first column for each mode).
- **Cond. Area**: as ϕ increases, condenser size requirements (A_C) are slightly reduced for the DOM columns, but slightly increased for the MOM columns. Moreover,

cDME-dc1 and cMMeOH-dc2 have larger condensers owing to the fact that these are the main product columns.

- **Reb. Area**: Optimal A_R increases with increasing ϕ for the MOM, but decreases for the DOM columns. Reboilers of the MOM are also larger than the corresponding reboilers in the DOM columns.

Recalling that costs are directly related to the physical parameters, new design parameters (D, N_S, A_C, A_R,) lead to new values of TDC, UTL, and TAC. **Figure 4.4** contains the results, shows the corresponding effect on design costs of due to the impact of ϕ on the physical design parameters. In all but the bottom-right subplot, optimal costs are shown for the DOM (blue), MOM (green) and for the overall (red) four-column design of **Figure 4.1(a).**

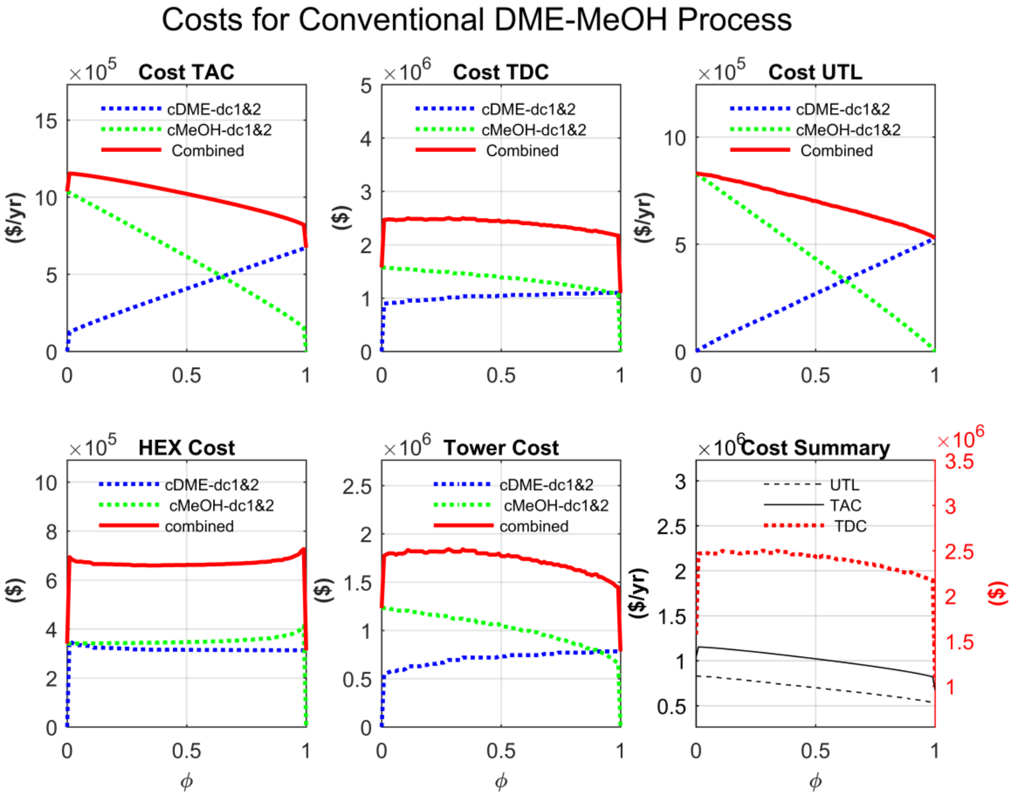

Figure 4.4 Optimal Costs for the conventional design process for variable ϕ. Curves for DOM, MOM, and also the cumulative costs of the overall design are shown.

In DOM (blue) the cost are for the condensers and reboilers of cDME-dc1 and cDME-dc2, while the corresponding columns for the MOM (green) being cMeOH-dc1 and cMeOH-dc2. Each subplot in the figure is discussed further below (for the $0 < \phi < 1$ range):

- **HEX Cost**: optimal heat exchanger costs at each value of ϕ are calculated from optimal the condenser and reboiler areas determined previously in **Figure 4.3**. Although individual heat exchanger areas and associated costs varied among the different columns of each operating mode, the total heat exchanger cost for both the DOM and MOM are nearly the same for all values of ϕ. The overall heat exchanger cost rises slightly anytime one mode is activate more than approximately 90% of the time, otherwise remaining relatively flat.

- **Tower Cost**: optimal distillation tower cost, including sieve tray cost and column shell cost, for the individual modes is highly sensitive to ϕ (owing to the previously shown sensitivity to **ϕ** of the underlying independent variables--D and N_T—used in determining tower cost). The opposing behaviour of the two mode's sensitivity to ϕ has a smoothing out effect on the overall tower cost for the four-column conventional design. Moreover, the shape (concave down) of the tower cost curve appears to be opposite that of the hex cost curve (which is concave up)—any reduction in heat exchanger cost with increasing ϕ is offset by an increase in tower cost.
- **Cost TDC**: TDC is the sum of tower and heat exchangers costs. Due to the tower costs being more than twice the heat exchangers costs, the overall shape of optimal TDC mirrors that of the tower costs. For DOM, TDC is insensitive to ϕ, but appears to decrease by a substantial amount for MOM in response to increasing ϕ .Total TDC for the whole convectional design overall shows little sensitivity to ϕ; showing only a minor decrease with values of ϕ greater than 0.5.
- **Cost UTL**: optimal total utility cost for the whole design decreases with increasing ϕ. UTL for DOM shows direct proportionality to ϕ, while being inversely proportional in the MOM case. Owing to UTL for MOM having a steeper slope than the DOM case, leading to the total UTL being monotonic in ϕ.

- **Cost TAC**: finally, optimal TAC for the whole conventional design is also monotonic in ϕ, reflecting that UTL has a much bigger impact on the TAC than does TDC. If more time is spent producing DME, the design ends up being more cost effective; that is, as ϕ increases, TAC for the overall conventional design decreases.
- **Cost summary:** this shows the relative magnitudes of each of the three main costs—TDC, UTL, and TAC—for the overall conventional DME and MeOH separation process. The value of TAC is approximately 1.2*UTL, and also approximately 2*TDC (in the range of interest:$0 < \phi < 1$). The utility cost appears to have the greatest influence on optimal TAC and also shows has similar response to ϕ.

It is important to reemphasize that each point on each of the curves (red) for combined costs is optimal and represents a design decision given anticipated operating breakdown in the two modes. Depending on the value of ϕ selected, entirely different designs (with at least one of D, N_A, N_B, Ac, and A_R being different) are required. To assess the behaviour of the optimum (at each ϕ value) we take the slope at each point and plot that against ϕ, the results of which appear in **Figure 4.5.** Using the Centred Finite Difference (CFD) approach, the slope is taken at each value of ϕ (excluding ϕ =0 and ϕ =1 points; so as to eliminate from the analysis the effect of the discontinuity between ϕ =0 and ϕ =.01, and between phi=0.99 and ϕ =1). Besides, as explained previously, the end points are not part of the same optimization as the points in the $0 < \phi < 1$ range.

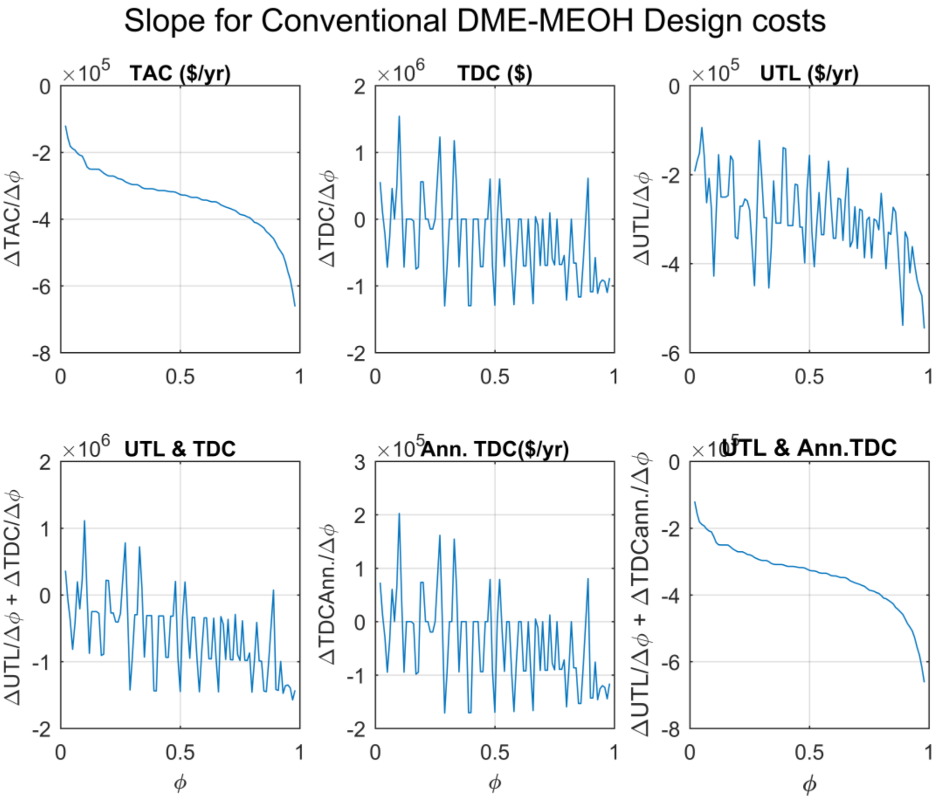

Figure 4.5 Slopes of various cost curves for the conventional process for 0< ϕ <1

TAC (top left) and UTL (top right) are indeed monotonic in ϕ: both always non-increasing with increasing ϕ, as shown by the slope values always having the same sign (for TAC) or being non-positive (for UTL). A closer look at the slope for TDC reveals that this cost is not monotonic in ϕ, indicated by the slope assuming different signs (both negative and positive) as ϕ increases. Further, this behaviour persists in the annualized TDC (bottom centre), the noisiness of which shows that the effect of ϕ on TDC is not always predictable, due in large part to the discrete part (N_A and N_B) of the optimization problem. Crucially, to further emphasize that each TAC value (for a given ϕ) is an optimum, an observation of the sum of the slopes of both the annualized TDC and UTL (bottom right)—the two variables that comprise TAC—reveals that their behaviour opposite. The micro level noise observed in these two variables is correlated. As ϕ is adjusted, if UTL moves in one direction, annualized TDC simultaneously changes by in the opposite direction, resulting in a non-decreasing TAC curve at the macro level, even though the function has lot of noise at the micro level. This noise is due to the discrete nature of the optimization. The crucial point to note is that TAC will still be at a minim even though TDC or UTL may increase at a given value of ϕ. This agrees with the known phenomenon in optimization of a distillation column: a compromise between capital cost (represented by TDC in this discussion) and utility cost must be made when attempting to minimize the total annualized cost of the column design.

4.3.3 Intensified Flexible Design

The proposed flexible design, in contrast to the conventional design, which uses *fixed* distillation equipment, allows for use of *multi-purpose* distillation equipment. This dual purpose nature is what gives this process intensification design approach the designation of “flexible”.

In this section, the results of the optimal design of the process depicted in **Figure 4.1 (b)** are presented. The approach followed is similar to the one in the previous section (4.3.2.2). It is again worth noting that here; too, the designs reduce to single-product case of section 4.3.2.1 for the special cases when $\phi=0$ (MeOH as the only product) and $\phi=1$ (DME as the only product). Where ϕ is the fraction of time in an operating year dedicated to operating in the DME Operating Mode (and 1- ϕ represents the [remaining] fraction of time spent in MeOH Operating Mode). Therefore, to motivate the discussion relating to the simultaneous production of both DME and MeOH, the analysis will again focus on the cases when$0 < \phi < 1$.

In this flexible design configuration, the same two distillation columns—f-dc1 and f-dc2—are used for both the DME Operating Mode (DOM) and MeOH Operating Mode (MOM). Optimal physical design parameters for varying values of ϕ are shown in **Figure 4.6**

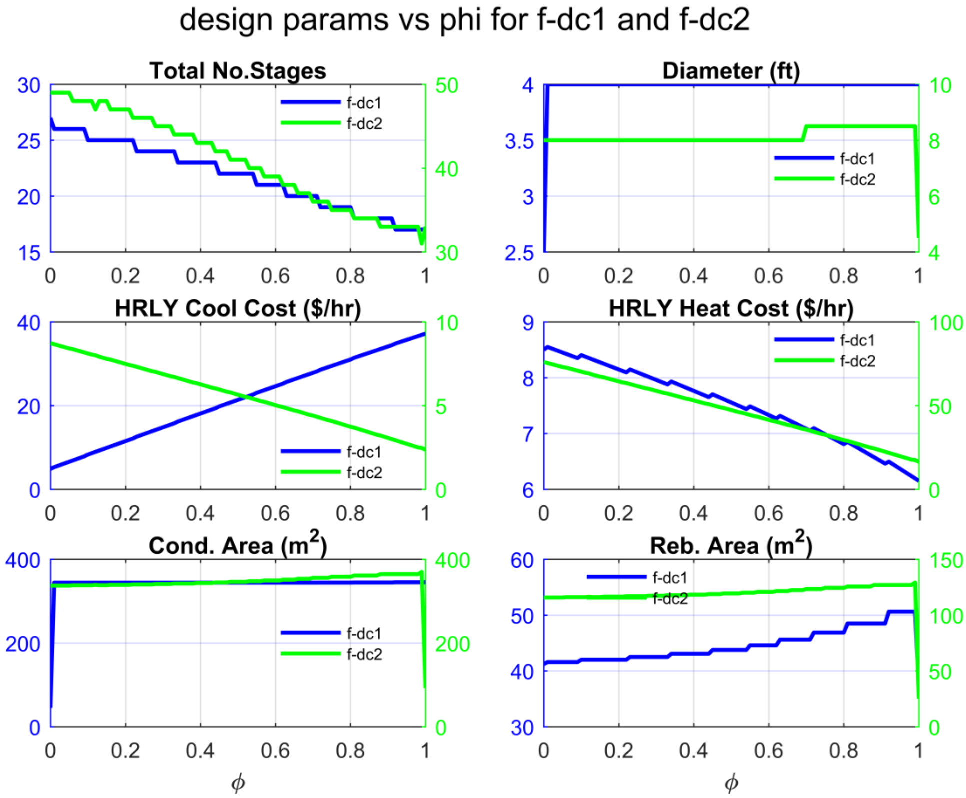

Figure 4.6 Summary of optimal physical design parameters plotted against ϕ for the flexible, multi-purpose design. Also shown are the cooling duty and heating duty hourly costs.

In order to better highlight the difference in behaviour between the optimal values of the two columns—f-dc1 (blue) and f-dc2 (green)—the variables of interest are plotted on separate vertical axes. The discontinuities shown as ϕ approaches 0 and as ϕ approaches 1 are due to reasons previously stated. The following observations are made from the charts (**Figure 4.6**) above, for the $0 < \phi < 1$ range:

- **Total No. Stages**: N_S for both f-dc1 and f-dc2 decreases as ϕ increases; that is, as more time is spent in DOM, the optimal total number of stages decreases. As more time is spent instead producing methanol, increasingly more stages are needed for the design to be optimal (that is, of minimum TAC). This observation agrees with the preliminary analysis of the simulation section (4.2) which showed that product MeOH requires longer distillation columns than that of product DME purification. Product DME is collected from f-dc1, while product MeOH comes out of f-dc2. For both columns, the same value of N_S is optimal for a range of ϕ values, as represented by the "stepped" nature of the plots. This range is far larger for f-dc1 than for f-dc2 at all ϕ values.

- **Diameter**: optimal diameter, D, for both columns is largely insensitive to ϕ, when both products are being made (for the $0 < \phi < 1$ range), that is, when designs capable of producing both DME and MeOH are being considered.
- **Cond**. **Area**: optimal condenser area, $A_{C,}$ for f-dc1 does not change as ϕ increases. This is due to the fact that the condenser that is optimal for DOM is large enough to handle MOM. Although this is an overdesign when in MOM, it is nonetheless the design that minimizes TAC—and is thus optimum. For f-dc2, the condenser size requirement increases only slight--by less than 4% for the entire range—with ϕ.
- **Reb**. **Area**: as ϕ increases, optimal reboiler area, $A_{R,}$ for f-dc1 is increases steadily, increasing by approximately 10% in the ϕ range. For f-dc2 the change of reboiler area with ϕ appears to be slight, increasing by about 5% in the entire $0 < \phi < 1$ range. Although both columns had nearly identical condenser areas across all values of ϕ, their reboiler areas are different, with f-dc2's reboiler being considerably larger than that of f-dc1.

- **HRLY Cool Cost and HRLY Heat Cost**: The optimal average hourly heating and cooling costs (which capture the directly related to the heating duty, Q_R and the cooling duty, Q_C) for both columns are highly sensitive to ϕ. This is the most obvious behaviour since the amount of time of operation directly impacts energy consumption. For f-dc2, both average heating and cooling costs fall with increasing ϕ. This is due to the fact that less product gets removed from f-dc2 as ϕ increases (more product is in fact removed from f-dc1).Furthermore, reboiler cost is an order of magnitude higher for f-dc2 as this column as the task of separating out all the water—which is higher boiling than both DME and MeOH—from the original ternary mixture..

Costs corresponding to the optimal physical design parameters determined above are provided in **Figure 4.7**. Optimal costs are provided for both columns—f-dc1 (blue curves) and f-dc2 (green curves)—of the flexible design.

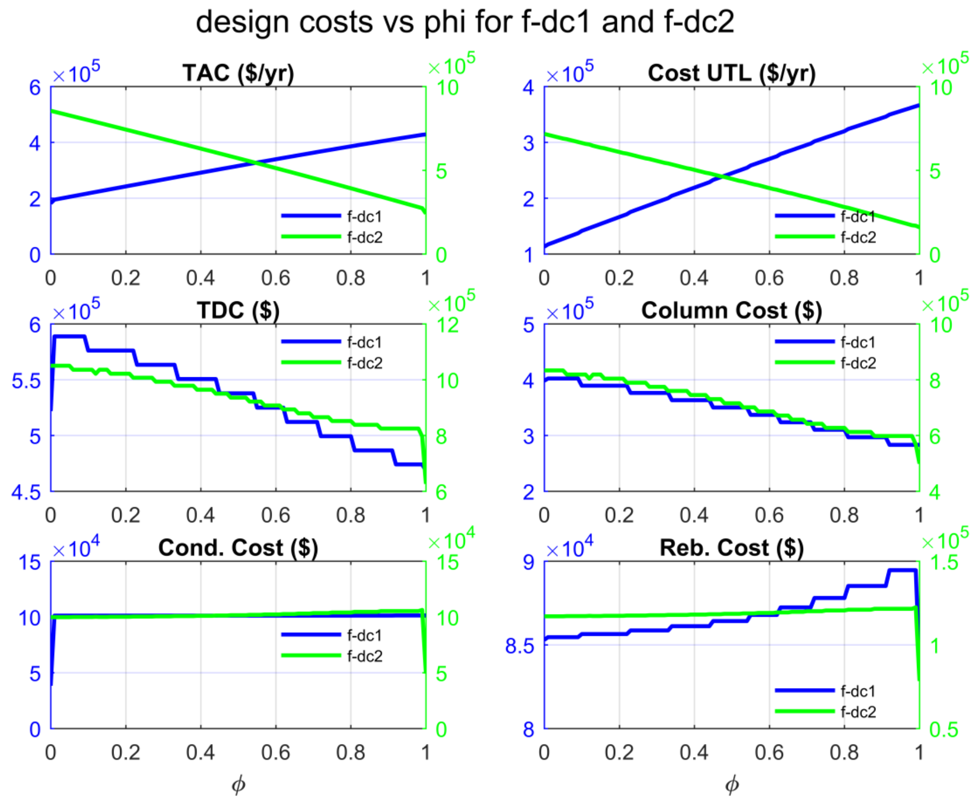

Figure 4.7 Summary of costs plotted against ϕ for the flexible, dual purpose design.

The following observations are made from the charts in the figure:

- **Cond. Cost & Reb. Cost**: condenser and reboiler costs are functions of their respective surface areas. As such, the observed behaviour mirrors that of the underlying heat exchanger areas discussed in **Figure 4.6.** Condenser cost (Cost$_C$) shows little to no sensitivity to ϕ for both f-dc1 and f-dc2. The reboiler cost (Cost$_R$) for both f-dc2 -, similar to the reboiler areas, show correspondingly little sensitivity to ϕ. But for f-dc2, optimal reboiler cost increases steadily with ϕ.
- **Column Cost**: the optimal column or tower cost (Cost$_{dc}$), which is a function of both number of stages (N$_S$) and column diameter (D), decreases with increasing ϕ for both f-dc1 and f-dc2. But because D remains unchanged with ϕ, the profile of

optimal $Cost_{dc}$ reflects mainly that of the Total No. Stages plot in **Figure 4.6**, indicating that $Cost_{dc}$ is dominated by N_S for all values of ϕ.

- **TDC**: the column's total direct cost comprises the column (tower cost) and the heat exchangers (condenser and reboiler) costs. Because the heat exchanger costs do not change substantially with changing ϕ, again the optimal TDC is dominated by the remaining constituent: column cost. The main observable trend is that as increasing more time is spent in DOM mode; the optimal total direct cost for both columns is reduced. This agrees with the observation made earlier regarding number of stages of columns for DME separation being substantially less than the number of stages required for columns of MeOH product separation. Specifically, DME separation requires thinner (smaller diameter), shorter (few stages) distillation columns compared to MeOH separation. These smaller DME columns therefore necessarily have comparably lower optimal TDC.
- **UTL**: the optimal utility costs for f-dc1 increases as more time is dedicated to DOM (increasing ϕ). At the same time, as less time is spent producing MeOH, the optimal utility costs for the MeOH product column (f-dc2) drop.
- **TAC**: total annualized cost is once more being dominated by the utilities cost, and as such its behaviour mirrors that of the constituent UTL. The annualized portion of TDC that constitutes the remainder of TAC appears to have a limited impact on optimal TAC for both f-dc1 and f-dc2.

A summary, in the form a table, of the main findings made thus far is provided in **Table 4.6**. Optimal design parameters for the flexible design process for the separation of both

DME and MeOH using two flexible, multipurpose distillation columns are provided for several select values of ϕ

Table 4.6 Summary of optimal results of the flexible design process for select values of ϕ

	ϕ	N_A	N_B	N_S	N_F	D	RR	BR	TAC ($/yr)
f-dc1	0	9	18	27	10	2.5	22.4	0.170	$ 180,000
	0.25	9	15	24	10	4	1.47	0.164	$ 260,000
	0.50	9	13	22	10	4	1.47	0.164	$ 320,000
	0.75	9	10	20	10	4	1.47	0.164	$ 380,000
	1	9	8	19	10	4	1.47	0.164	$ 430,000
f-dc2	0	40	9	49	41	8	0.811	7.28	$ 860,000
	0.25	36	10	46	37	8	0.826	7.35	$ 720,000
	0.50	31	10	41	32	8	0.859	7.51	$ 570,000
	0.75	25	11	36	26	8	0.921	7.81	$ 430,000
	1	22	11	33	23	6	1.46	0.49	$ 240,000

We can verify that in deed the values for the TAC for f-dc1 and f-dc2 at ϕ=0 and ϕ=1 in **Table 4.6** for the flexible design are *exactly the same* as those found in **Table 4.5** for the conventional design, both corresponding to special cases where only one product is made.

In **Figure 4.2** , the so-called direction of optimality was briefly discussed. In the figures that follow, attempts to answer that same question of the directionality of the optimum are undertaken. The figures also serve as proof that the entire feasible space was explored in search of the optimal design parameters for a flexible distillation design. The feasible points at each ϕ (since a large number of ϕ values exist in the range$0 < \phi < 1$, only a few values—0, 0.25, 0.75 and 1) are chosen to illustrate the point). The colour maps shown forms the foundation of the exhaustive search discrete optimization framework (in which every feasible point is visited when searching for the optimum) that is employed in this work. In each figure, the point shown in bold red represents the optimum value (for the given ϕ value). It is important to note that the optimal point is chosen as the point whose TAC is the lowest for all feasible points surveyed.

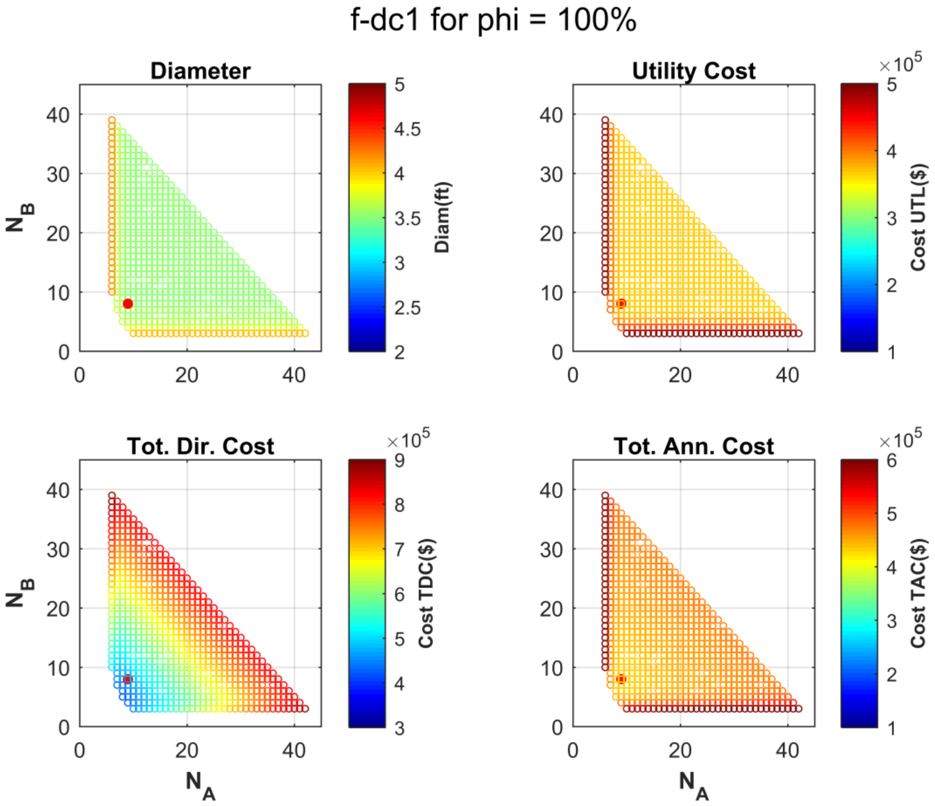

Figure 4.8 Cost summary for phi=1 for the flexible design, for f-dc1

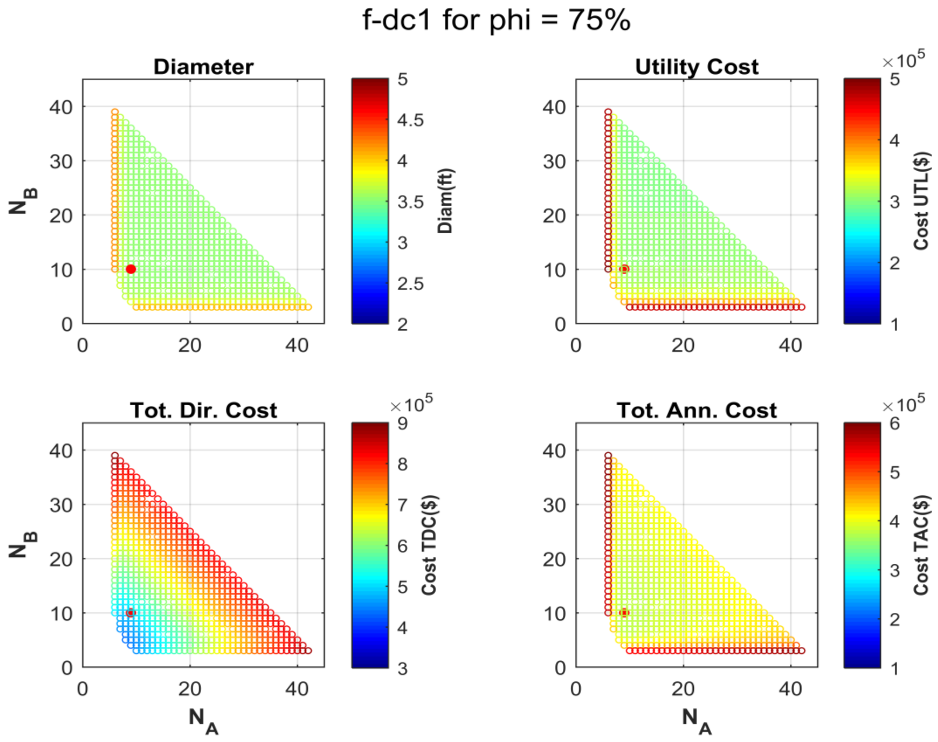

Figure 4.9 cost summary for ϕ =0.75 for the flexible design, for f-dc1.

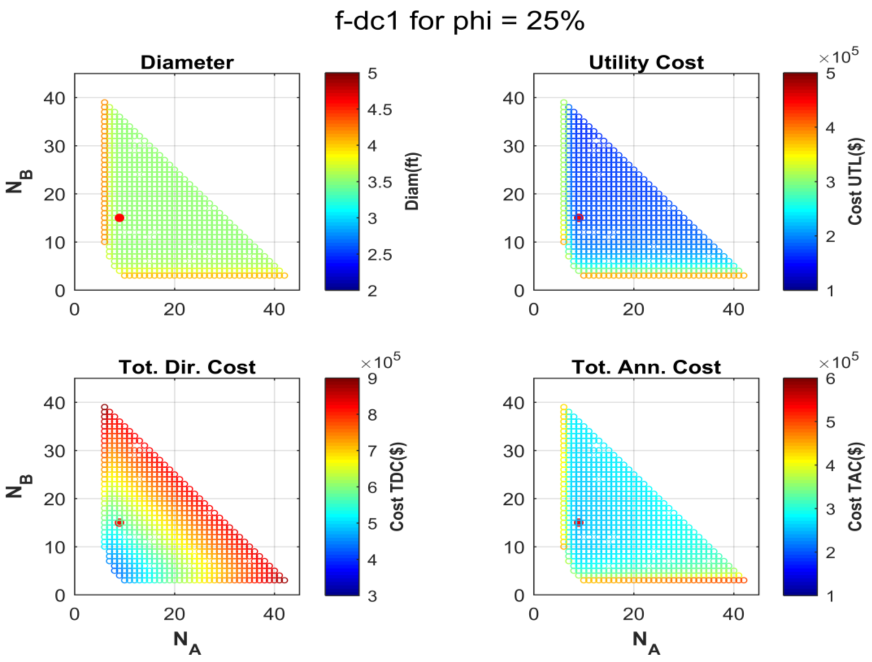

Figure 4.10 cost summary for ϕ =0.25 for the flexible design, for f-dc1.

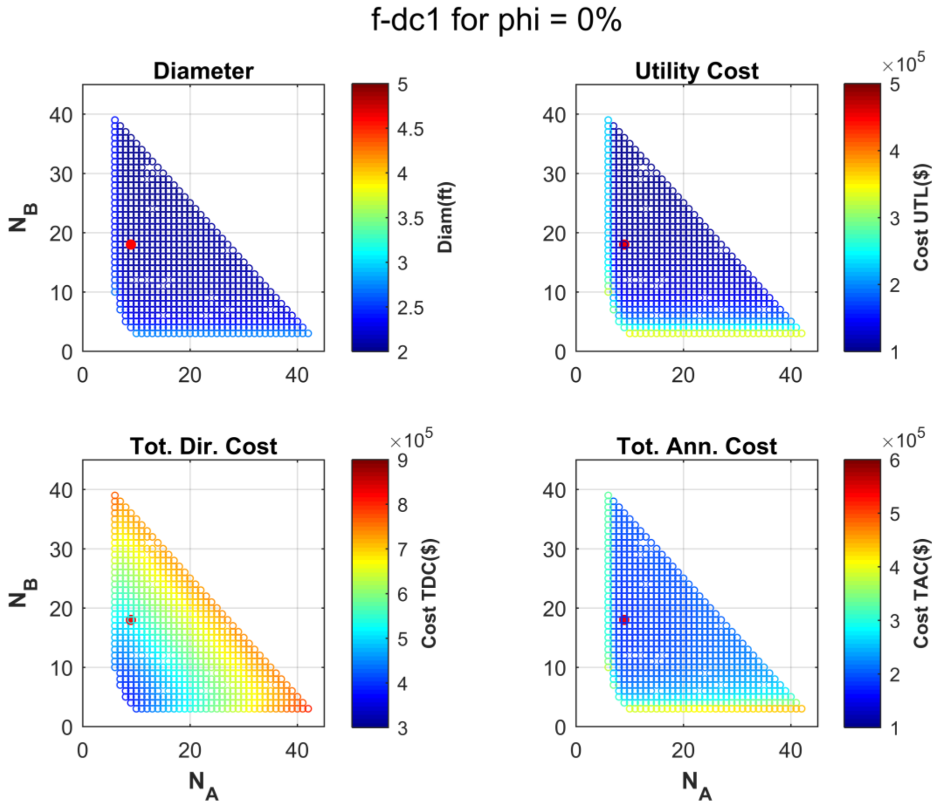

Figure 4.11 cost summary for ϕ =0 for the flexible design, for f-dc1.

The preceding plots--**Figure 4.8, Figure 4.9, Figure 4.10,** and **Figure 4.11**—show how changing the value of ϕ affects column optimal diameter (D), optimal utility cost (UTL), optimal total direct cost (TDC), and optimal total annualized cost (TAC) for the first column (f-dc1) of the flexible design. In each figure, the four variables of interest are plotted against the no. of stages above the feed (N_A) and no. of stages below the feed (N_B). The figures correspond to data displayed in the top half of **Table 4.6** It is immediately obvious that the optimal values (red dots), which depend on ϕ, change from figure to figure; and the direction of the optimum is also visible in each subplot. The limits on the colour bars are kept constant across the figures to highlight the important changes as ϕ is adjusted from 1 to 0. The observed changes corroborate the findings depicted by the TAC plot in **Figure 4.7:** wherein it was shown that f-dc1 TAC decreases with decreasing ϕ.

The next four figures are for the second column (f-dc2) of the flexible design. The figures are ordered by increasing value ϕ (from 0 to 1).

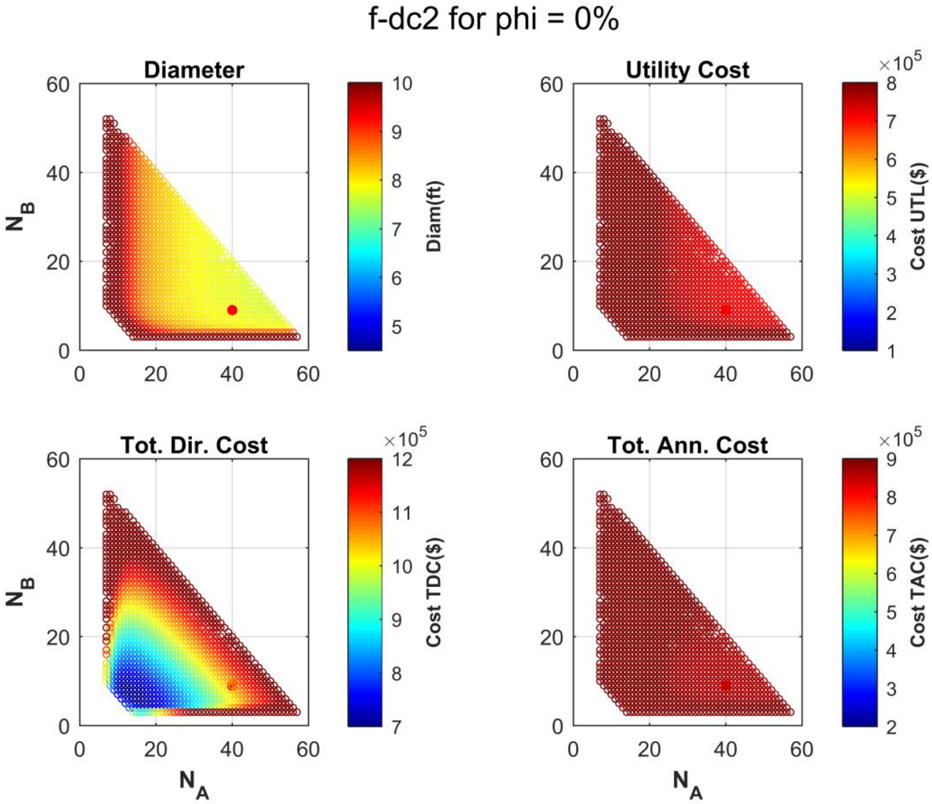

Figure 4.12 cost summary for ϕ =0 for the flexible design, for f-dc2.

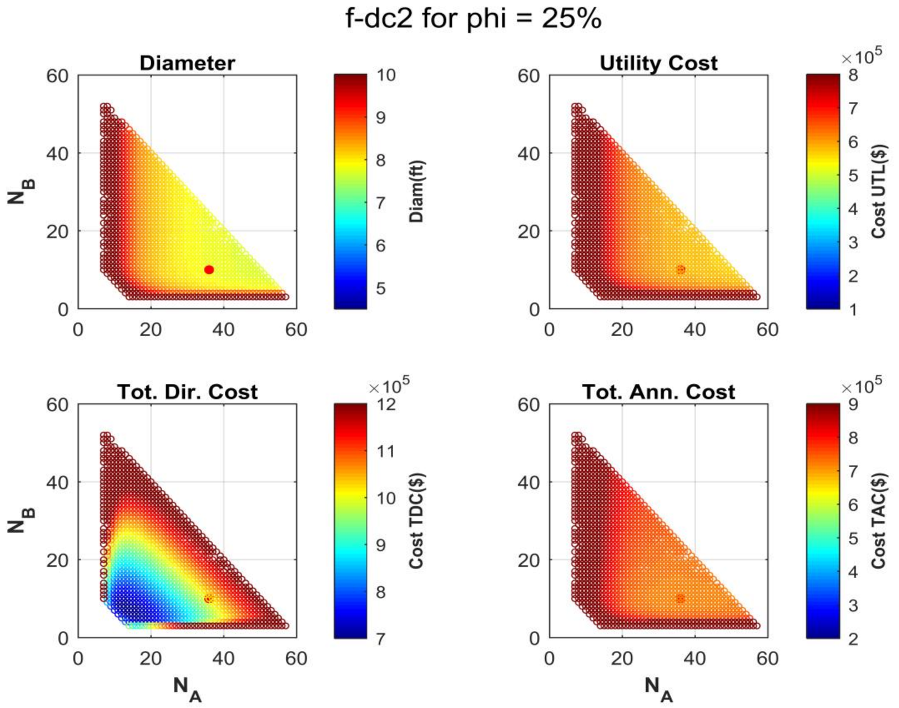

Figure 4.13 cost summary for ϕ =0.25 for the flexible design, for f-dc2.

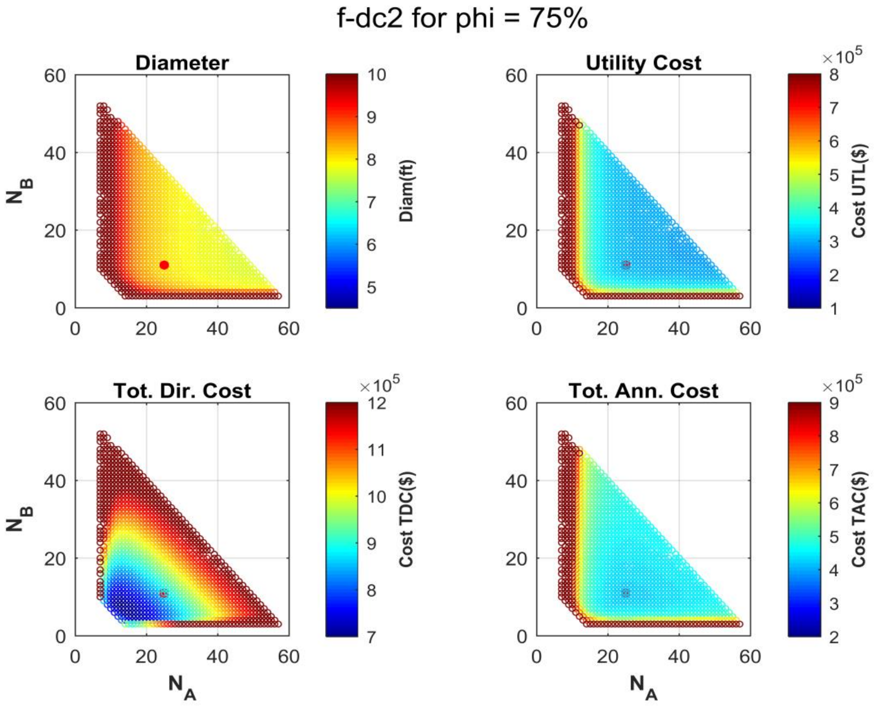

Figure 4.14 cost summary for ϕ =0.75 for the flexible design, for f-dc2

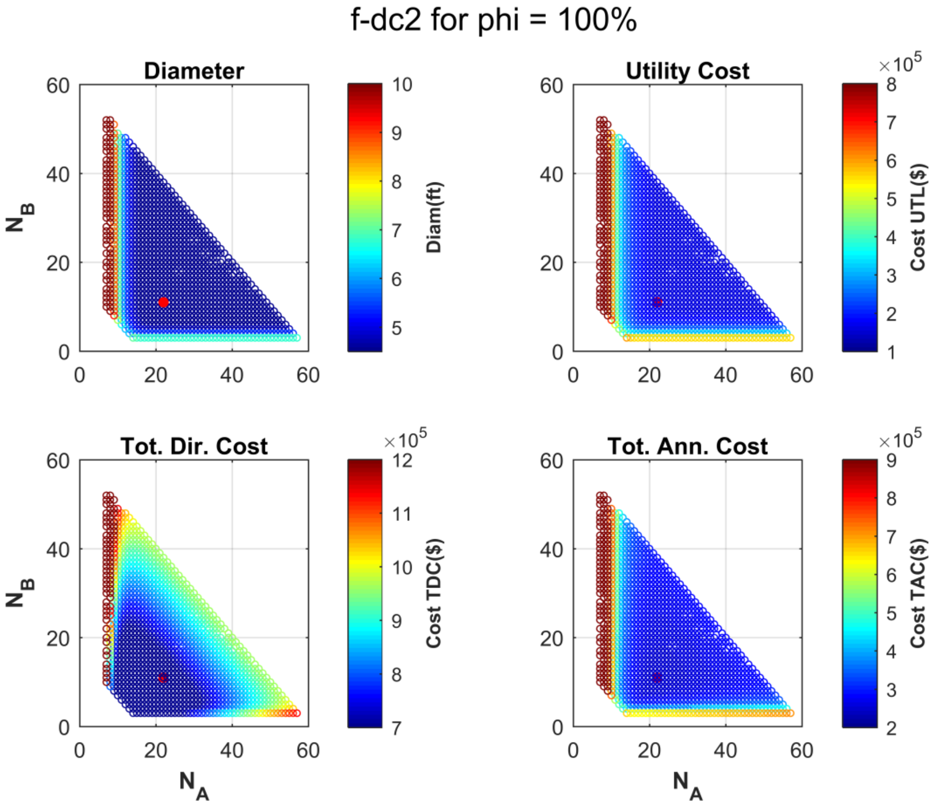

Figure 4.15 cost summary for ϕ =1 for the flexible design, for f-dc2.

The findings from the preceding set of figures--**Figure 4.12, Figure 4.13, Figure 4.14,** and **Figure 4.15**—again confirm the findings of the latter half of **Table 4.6.** Specifically, it can be seen that: the optimal (minimum)TAC for the second column of the flexible design decreases as ϕ increases, as do the corresponding TDC and UTL. This again agrees with the previous finding of **Figure 4.7**.

In order to determine the overall total costs for the flexible process, the cost of the two columns are summed up at each ϕ. The final results are illustrated in **Figure 4.16**.

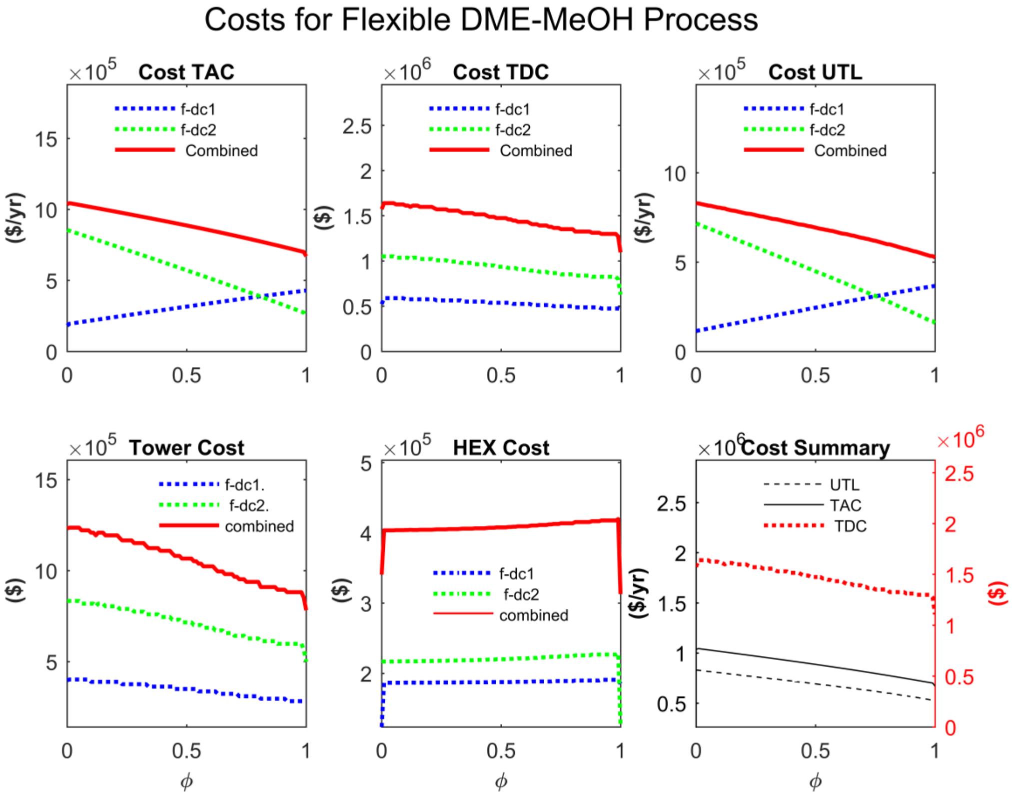

Figure 4.16 Costs for the flexible, dual purpose design for variable ϕ

The behaviour of the individual column costs has already been discussed. The task at hand is to highlight the findings regarding the total (combined; red) costs for the entire flexible design process, focusing as usual on the range of interest: $0 < \phi < 1$. TAC, which decreases with increasing ϕ, is heavily influenced by UTL, with TDC having only a light effect on optimal TAC. In addition, all three major costs—TAC, TDC and UTL—appear to always decrease with **ϕ.** Heat exchanger cost once again shows almost no sensitivity to changing ϕ. Tower cost appears to have the greatest influence on TDC, with TDC having a nearly identical profile to that of the tower cost. Like all other costs, excluding the heat exchanger cost, tower cost appears, in general, to always decrease with ϕ. The apparent monotonic behaviour of the cost curves is explored further in **Figure 4.17** below.

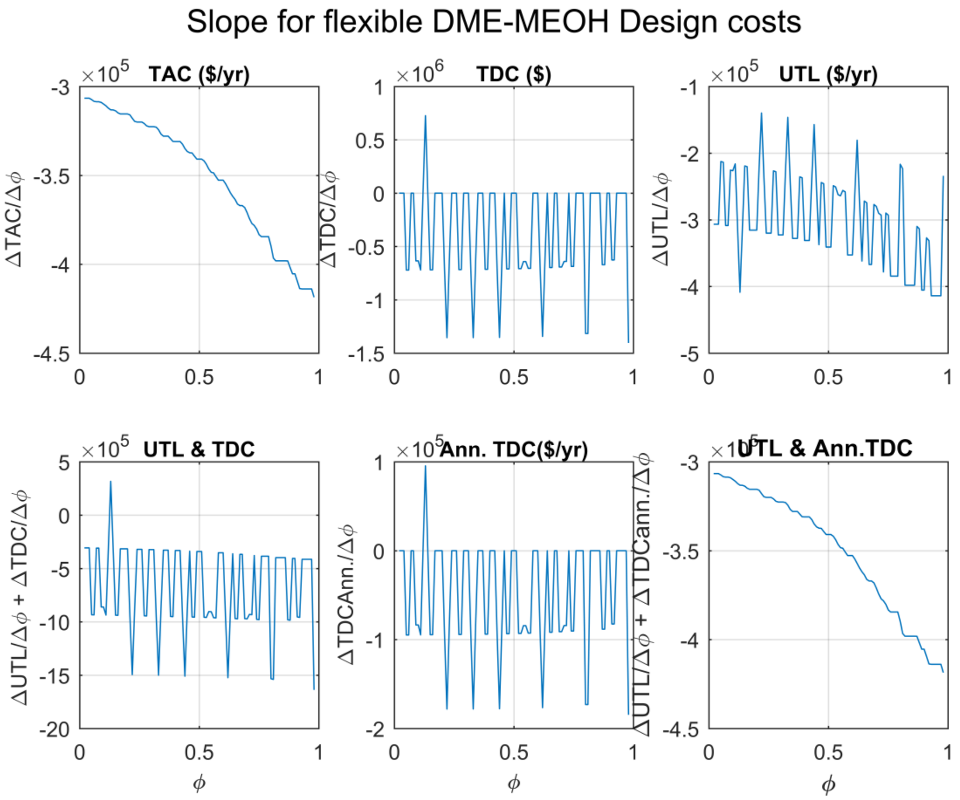

Figure 4.17 Slopes of flexible DME-MeOH design costs curves. Centred Finite Difference (CFD) approach is used to approximate the slope at each discrete point. All costs are monotonic in ϕ.

Each cost's slope shows that the cost are always decreasing for TAC (or equivalently, for UTL + annualized TDC) and UTL. Though TDC and UTL are quite noisy, the noise is once again negatively correlated, leading to a relatively noise-free optimum TAC (or UTL + annualized TDC) curve at the macro level.

In summary, for the flexible design: when more time is spent in DME operating mode, the design has the lower costs (TAC, TDC, and UTL). The TAC summaries are provided in the table below.

Table 4.7 Summary of optimal overall (both columns) TAC for the flexible design for select values of ϕ

ϕ	TAC ($/yr)
0	$ 1,040,000
25%	$ 970,000
50%	$ 890,000
75%	$ 800,000
100%	$ 670,000

4.3.4 Proposed Flexible Design vs Conventional Design

At the outset, a hypothesis was put forward, which stated that applying techniques of process intensification to the separation of DME and MeOH by repurposing distillation equipment could yield cost savings on proposed innovative plant design. In this section, this hypothesis is proven true: the process intensification carried out during the design of separation processes for DME and MeOH purification plants reduces the TAC design. Specifically, the flexible design has lower optimal TAC over all expected operating regimes; that is, for all values of ϕ in the interval $0 < \phi < 1$. These important findings from the optimization process are presented in **Figure 4.18.**

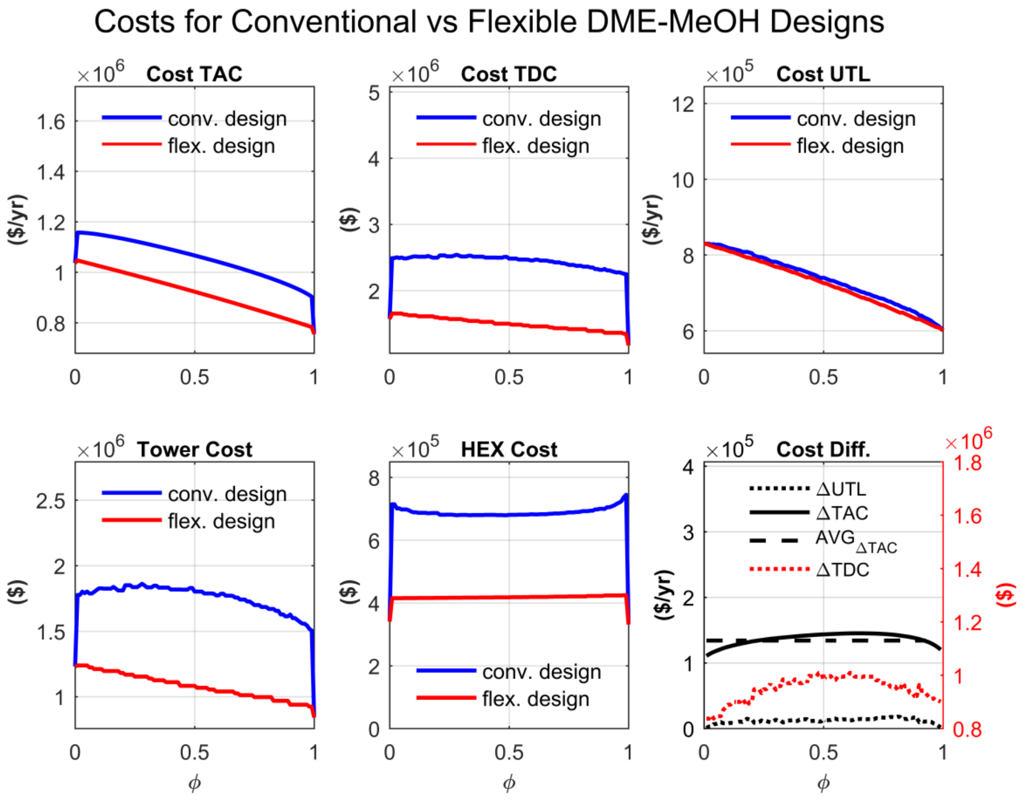

Figure 4.18 Cost comparison between conventional and flexible designs for the separation of DME and MeOH for variable ϕ

Each subplot shows a graph of the conventional process alongside that of the proposed flexible design. The TAC, TDC, UTL, and tower and heat exchangers costs are compared for the two designs. The final plot is a graph of the difference (or cost savings) between the cost of the conventional design and that of the flexible design.

Table 4.8 Average cost savings of flexible design over conventional design, for 0 < ϕ < 1

	TDC	UTL	TAC
Avg. Savings (Conv. - Flex.)	$ 930,000	$ 10,000	$ 130,000

A majority of the cost savings results from a reduction in TDC, as equipment is removed and plant foot print is reduced. The UTL savings are not significant, (with all costs having been rounded to the nearest $10,000). As such, the bulk of the TAC savings are due to savings is capital cost.

Another critical component of the flexible design being proposed is its ability to seamlessly switch between two very different feeds, with little cost penalty. An important distinction is needed when talking about the switching of feeds; specifically, we are not talking here about the *marginal* TAC change when increasing ϕ from 0 to 0.01. The effects of *dynamic transitions* are not being considered at this point, and are considered beyond the scope of this work. The findings are summarized in **Table 4.9**. Compared to the conventional design, over 90% in TDC and TAC for the conventional design is saved by using the proposed flexible, multi-purpose distillation column design. The cost penalty is taken as the difference between (1) the cost at the first value where ϕ >0 (such as ϕ=0.01), and (2) the cost at ϕ=0.

Table 4.9 Cost penalty for product portfolio diversification for both conventional and flexible designs

	TDC Pen.	***UTL Pen*.***	***TAC Pen.***
Conv. Design	$890,00	-	$120,000
Flex. Design	$70,000	-	$10,000
Difference	$830,000	$0	$110,000

*UTL Penalty calculated is less than $1000 for each case

4.4 Summary and Conclusion

In this section, the results of the proposed process intensification scheme, namely, the optimal design of a flexible distillation sequence for the purification of DME and MeOH are presented. First, a review of the process is presented in a simplified process flow diagram, showing key separation processes, which are the focus of the optimization. Following the review of both designs for the conventional and proposed cases, simulation results are presented.

The next step involves presenting the results of the preliminary optimization of the stand-alone conventional processes, in which one feed is processed to yield one major product. Another layer of complexity is then added: the results of the optimized conventional process capable of producing both products using individual separation trains are presented. the key independent variable of interest, labelled **ϕ, is** the amount of time spent in each of the two modes of production—DME Operating Mode (DOM) and MeOH Operating Mode (MOM). Results from the optimization, showing the effect of varying ϕ on optimal design cost (on a Total Annualized Cost basis) and related design variables of the conventional design, are presented next.

Following a thorough review of the optimal design for the conventional process, the findings from the proposed flexible, intensified design are subsequently presented. In a similar manner to the conventional design case, the role of ϕ on optimal TAC of the two-column intensified design is brought to the fore.

Finally, a cost comparison is made, the results of which support our original hypothesis: process intensification of the DME-&-MeOH separation process could lead to substantial cost savings. Importantly, not only does the proposed flexible design yield reduced optimal cost (TAC) for all values of ϕ ($0 < \phi < 1$), but it also proves to be more adept—that is, showing a lower cost penalty as a result of being flexible—at adjusting to changes in the anticipated values of ϕ, when compared to the conventional design.

Chapter 5 CONCLUSION & RECOMMENDATIONS

5.1 Summary

The focus of this work has been on the realization of a novel process intensification scheme—the design of a flexible, intensified distillation process for the separation of DME and MeOH. An original and unique approach, aimed at exploiting the hidden benefits (cost savings) that can be reaped from reducing a plant's installed equipment volume and overall footprint, is embarked upon in solving the design optimization problem. The proposed trimming of installed equipment is realized by essentially repurposing all of the distillation equipment used in one separation process (DME) for use in another (MeOH). In order to realize such a flexible design, capable of meeting separation requirements for two distinct processes, rigorous design optimization is undertaken.

Relevant background information on DME and MeOH distillation processes is first reviewed, before the individual processes are modelled and simulated in. Aspen Plus, with the principal goal being to meet purity specifications 99.95 wt. % and 99.85 wt. %, respectively for DME and MeOH product. Aspen Plus's DSTWU shortcut column is used a starting point in the simulation, in order to establish lower bounds on the number of stages and reflux ratio required to achieve the specified purity specifications. Following the preliminary bounding step, Aspen Plus's RADFRAC—a modelling tool for more rigorous simulations of distillation processes—is used to obtain higher fidelity results. These latter results of the RADFRAC simulations, which include physical (such as, number of stages, feed stage, column diameter) as well as operating (condenser and reboiler heat duties, reflux and boil-up ratios) parameters for the distillation processes are used as initial guesses in the subsequent optimization phase, implemented with the aid of Aspen Plus's "design spec" and "vary" tools. The "design spec" and "vary" tools are used to enforce constraints on the distillate purities of each column in the optimization step. The design-spec-vary combination solves nonlinear system of equations for a distillation column of specified number of stages and feed location. An analysis on various combinations of number of stages and feed tray location is carried out using Aspen Plus's "sensitivity analysis" tool. Last but not least, all combinations are evaluated for total annualized cost. Lastly, to determine the design with the lowest TAC, an algorithm, implemented in MATLAB, is furnished.

5.2 Recommendations for Future Work

5.2.1 Alternate Design Configuration

Throughout the course of this investigation a given sequence of distillation columns has been used. The process intensification is derived from a set sequence of distillation columns for the conventional design case, and as such, the question of column sequencing was not addressed. A future analysis examining the potential marginal benefit (that is, further cost reduction) of switching the columns between the two operating modes could be undertaken. A schematic of the envisaged alternate configuration that might be explored is illustrated by Figure 5.1 below.

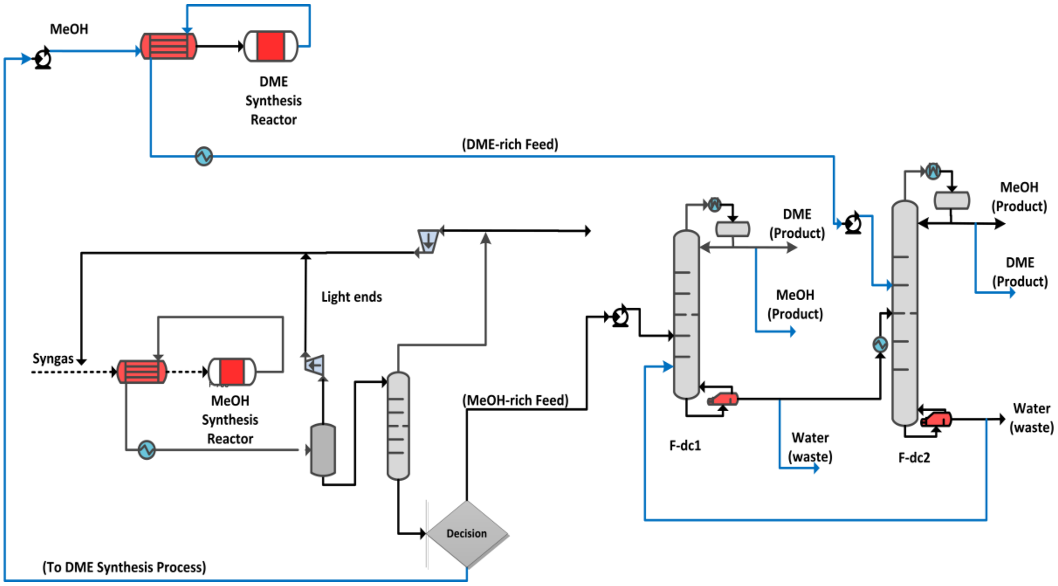

Figure 5.1 Alternate Design

In Figure 5.1 above, as in the original proposed intensified design, the decision block determines the fate of the crude methanol stream just like before; however, the effluent of the DME synthesis reactor now enters the distillation section of the process flow diagram at an entirely different location (column number and feed stage number) than in the MeOH operating mode.

When the process is in MEOH operating mode, syngas first enters the MeOH synthesis reactor, is flashed and gets passed through a stripper, before entering the F-dc1 column in the distillation train. The more volatile DME component is collected as distillate at the top while the MeOH and Water mixture continues to the F-dc2 column from which pure MeOH is collected as product in the distillation and the waste water removed as bottoms. Conversely, in the DME operating mode, syngas enters the MeOH synthesis reactor as before, goes through the flashing and stripping processes as before, is then routed to the DME synthesis reactor. Crude DME (DME synthesis reactor effluent) is sent first to the F-dc2 column—the crude DME feed enter s on a newly specified feed location on F-dc2. In F-dc2, high purity DME is removed as distillate while the MEOH –Water mixture leaves out the bottoms and continues to the F-dc1 column, which is the second column in the sequence in this new configuration. High purity MEOH product is removed as distillate while the waste water leaves as the bottoms from the F-dc1 column.

The motivation behind the alternate column configuration comes from the realization that in the original configuration of the proposed intensified design, the product column experiences the higher flow rates during each of the two operating modes. That is, during DOM, column F-dc1 is the column with the higher flow rates while in MOM column F-dc2 experiences the higher flow rates. From this realization, it is evident that a possible opportunity may exist where in this peculiar behaviour is exploited and further design optimization is realised. A thorough and more rigorous analysis of this scenario could therefore be of potential benefit/is worth exploring for potential cost savings.

5.2.2 Dynamics Consideration

Thus far all analysis and simulation decisions have been carried out under steady-state operation assumptions. However, in order to truly bring the results of the proposed analysis closer to industrial applicability an analysis of the various dynamic aspects of the processes would prove useful. The dynamics effects that we would be concerned with would mostly affect the separation section of the process in (Figure 3.2). Of special concern would be the effects of start-up and shut-down (and purging) of distillation train between operational periods and modes. So far the effects of transitions have been assumed negligible, if a sufficiently long operational time during each mode is assumed.

The consideration of process dynamics naturally leads to the requirement of solving a dynamic optimization problem--the solution of which will yield optimal design and control decisions required for cost minimization.

5.2.3 Stochastic Effects

The entirety of the approach employed in formulating and solving the problem has followed a deterministic approach. However, realizing that there exists inherent uncertainty in one of the key optimization variables—ϕ, the fractional amount of time spent operating in DME mode—there arises a need for a probabilistic approach to the problem.

The current design assumes, to an extent, that ϕ can be somehow known (forecast), but what happens when ϕ is truly uncertain? This gives rise to a design under uncertainty problem; the solution of which could involve application of the classic “two-stage stochastic optimization” framework.

Alternatively, we could realize the exhaustive search approach used in the current formulation lends itself well to the application of statistical techniques to the existing large data set. For example, we could employ a probability distribution to characterise the behaviour of the uncertain parameter,ϕ, in a so-called distribution fitting approach. The new, non-deterministic objective function would then become a minimization of the expected value of TAC:

$$\text{Minimize} \qquad \mathrm{E}(TAC)$$

5.3 Conclusion

The successful design and optimization of a novel, intensified, flexible separation process for DME-MeOH production under multi-period operation consideration is performed. Results show that, regardless of the length of the operating period in each mode, denoted as ϕ, an optimal intensified design will have lower Total Annualized Cost compared to current designs employing two separate distillation trains for DME and MeOH separation. While opportunities exist for further fine tuning of the propped process design including, considering different distillation column sequencing, accounting for dynamic effects, and employing probabilistic methods, the results so far obtained provide a basis for industry personnel and other researchers to explore the potential benefits of applying process intensification to conventional distillation columns for DME and MEOH separation applications.

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