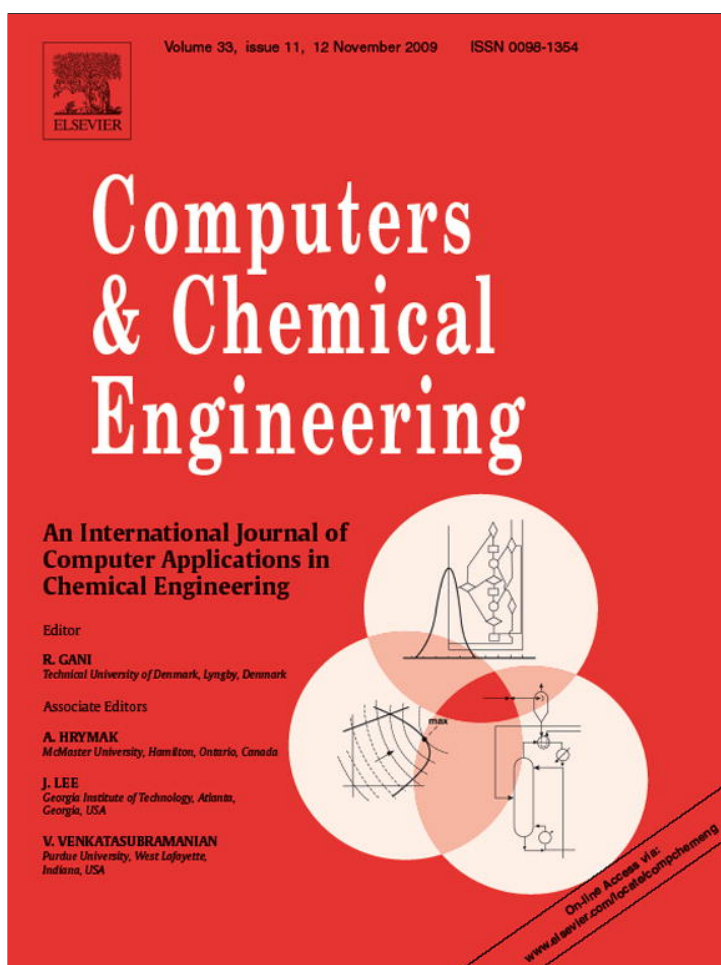


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## Design and optimization, using genetic algorithms, of intensified distillation systems for a class of quaternary mixtures

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## ABSTRACT

The design and optimization of a coupled multicomponent distillation system is a non-linear and multivariable problem. The complexity of this kind of problem results in high solving difficulty. This paper addresses the application of genetic algorithms to the optimization of intensified distillation systems for quaternary distillations. We used a multiobjective genetic algorithm with restrictions coupled to the Aspen Plus<sup>TM</sup> process simulator for the evaluation of the objective function. Several mixtures to test the effect of relative volatilities of feed mixtures on energy consumption, second law efficiency, total annual cost and theoretical control properties have been studied. Numerical performance shows that this design tool is robust and suitable for the design of coupled multicomponent distillation sequences.

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### 1. Introduction

Process intensification (PI) is an area presently receiving considerable attention in chemical engineering. Stankiewicz and Moulijn (2000) and Stankiewicz and Drinkenburg (2004) provide a definition of PI, comprising novel equipment, processing techniques and process development methods that, compared to conventional ones, offer substantial improvements in (bio)chemical manufacturing and processing; they also provide an extensive description of a PI toolbox, ordered along two dimensions: equipment and processing methods. This goal and others, especially the need to “maximize mass, energy, space and time efficiencies” and the concept of “output-pulled vs. input-pushed” are intimately related to a current emphasis on “green chemistry” in chemical technology (Malone, Huss, & Doherty, 2003). Distillation is responsible for about 3% of total U.S. energy consumption, more than 90% of all product recovery and purification separations in the U.S., and more than 95% of chemical industry consumption worldwide. Data from the United States Department of Energy indicate that distillation columns in the U.S. consume 5.07 million TJ per year. To get an idea of how large this number is, note that the 439 nuclear power plants

in operation worldwide have a total net installed capacity of 372 GW (Plesu, Bonet, Plesu, Bozga, & Galan, 2008). Distillation processes are highly energy-consuming systems, and any small improvement in distillation can provide huge energy savings.

Thermal coupling has been used in the design of multicomponent distillation systems to significantly reduce both energy consumption and capital costs over conventional simple column configurations. The dividing wall column with thermal coupling was first patented by Wright (1949). Petlyuk, Platonov, and Slavinskii (1965) introduced the thermal coupling for ternary distillations and presented a fully thermally coupled configuration (Petlyuk column). There is a considerable amount of literature analyzing the relative advantages of thermally coupled schemes for ternary separations (Finn, 1993; Hernández & Jiménez, 1996; Hernández & Jiménez, 1999; Premkumar & Rangaiah, 2009; Schultz et al., 2002; Sotudeh & Shahraki, 2008; Tedder & Rudd, 1978; Triantafyllou & Smith, 1992, among others). Specifically, the thermally coupled dividing wall column (DWC) has been successfully used in many industrial separations for ternary mixtures (Olujić et al., 2003). Thermally coupled distillation sequences (TCDS) are an example of process intensification (Jantes-Jaramillo, Segovia - Hernández, & Hernández, 2008).

The Petlyuk scheme is equivalent to the dividing wall column. The DWC offers the possibility of both energy and capital cost savings. Capital cost savings result from a reduction in quantity of equipment (i.e., one shell instead of two in the case of the Pet-

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**Nomenclature**

LF1	liquid phase interlinking flow, kmol h <sup>-1</sup>
VF1	vapor phase interlinking flow, kmol h <sup>-1</sup>
Q	reboiler heat duty, kW
TAC	total annual cost, USD

*Greek symbols*

$\gamma$	condition number
$\eta$	second law efficiency
$\sigma^*$	Morari resiliency index

lyuk column). There are also indirect benefits: a DWC requires less plot area, and, therefore, shorter piping and electrical runs. Flare loads are reduced because of the lower heat input and smaller fire-case surface, leading to a smaller flare system. Amminudin, Smith, Thong, and Towler (2001) noted the industrial acceptance and commercialization of DWC by organizations such as BASF AG, M.W. Kellogg (together with BP, later known as BP Amoco), and Sumitomo Heavy Industries Co. together with Kyowa Yuka. Linde AG constructed the world's largest DWC for Sasol, an estimated 107 m tall and 5 m in diameter (Schultz et al., 2002). In 2004, Adrian et al. reported that BASF operates about 30 DWCs worldwide in their plants. Recently, efforts have focused on finding new thermally coupled configurations based on energy savings as well as operability.

There has been some work on extensions toward the design of coupled systems for mixtures of more than three components (e.g., Agrawal, 1996; Blancarte-Palacios, Bautista-Valdés, Hernández, Rico-Ramírez, & Jiménez, 2003; Chrsitiansen, Skogestad, & Lien, 1997; Calzon-McConville, Rosales-Zamora, Segovia-Hernández, Hernández, & Rico-Ramírez, 2006; Errico, Rong, Tola, & Turunen, 2008; Mascia, Ferrara, Vacca, Tola, & Errico, 2007; Rong & Kraslawski, 2003; Rong, Kraslawski, & Nystrom, 2000). Optimal synthesis and design of multicomponent distillation processes are usually performed in a search space, which excludes the considerations of complex thermally coupled distillation schemes. Rong, Kraslawski, and Nystrom (2001) have proposed a shortcut design procedure for the design of multicomponent thermally coupled distillation columns. In 2006, Rong and Turunen proposed a process intensification method for systematic synthesis of new distillation systems with fewer than N-1 columns for an n-component mixture. The method is illustrated for quaternary distillations. The authors took advantage of simultaneous thermal coupling and heat integration as a process intensification strategy for the systematic synthesis of new intensified distillation systems for multicomponent separation. This provides opportunities for equipment integration and intensification. It was practical for a prefractionator to be incorporated into another column to share a single column shell. One simple way to accomplish this is through a dividing wall. It is important to highlight that the synergy of mass and heat transfers by simultaneous thermal coupling and heat integration enables equipment integration and intensification. It is equipment integration and intensification that ultimately produces the new intensified distillation systems, using a dividing wall, for separation of multicomponent mixtures. These configurations should result in savings in both energy and capital costs. Studies must be done on the new intensified systems relating to design, optimization and dynamic behavior because there is little literature on the use of dividing wall columns for multicomponent separations.

The optimal design of coupled systems for separation of multicomponent mixtures is a non-linear and multivariable problem, and the objective function used as optimization criterion is generally non-convex with several local optimums. However, the task

is complicated and is likely to fail to achieve convergence. Recent years have seen increased development and application of global optimization strategies in many areas of chemical engineering. Global optimization methods can be classified as deterministic or stochastic. The first class offers a guarantee of finding the global optimum of the objective function, provided that the objective function is convex. However, strategies in this class often require high computational time (generally more time than stochastic methods), and, in some cases, problem reformulation is necessary. The use of rigorous design and thermodynamic models leads to very large non-convex models, which are very difficult to converge. Moreover, taking into account structural and design decisions, such as the existence of stages, columns, condensers and reboilers, leads to the inclusion of integer variables further increasing the difficulty of solving the model. Finally, additional convergence problems are generated when discontinuous functions, such as complex cost functions, are introduced in the model. Efforts towards the optimal design of TCDS schemes for separation of ternary mixtures in the area of deterministic methods are present in the work of Dünnebier and Pantelides (1999), Yeomans and Grossmann (2000), and Caballero and Grossmann (2001). For a multicomponent mixture, the problem is clearly more complicated, as the combinatorial nature of the system results in a set of several structures that are significantly more complex to solve. There are a significant number of variables that must be determined in order to develop the complete design of a multicomponent thermally coupled distillation sequence. Despite the previous work of Rong and Turunen (2006), where the authors propose a complete set of alternatives to the separation of four components in DWCs, there is a lack of rigorous design methodology for this kind of structures. In this context, stochastic optimization methods are playing an important role because they are generally robust numerical tools that present a reasonable computational effort in the optimization of multivariable functions, are applicable to unknown structure problems, require only calculations of the objective function, and can be used with all models without problem reformulation (The & Rangaiah, 2003). In the case of stochastic optimization, there are methods known as genetic algorithms (GA), which are part of the wider field of evolutive algorithms. These algorithms were first proposed by Holland (1975) to solve optimization problems. Genetic algorithms are stochastic methods based on the idea of evolution and survival of the fittest. Genetic algorithms have several features that make them attractive for solving optimization problems with modular simulators, where the model of each unit is only available in an implicit form (black-box model). First, due to the fact that they are based on a direct search method, it is not necessary to have explicit information of the mathematical model or its derivatives. Second, the search for the optimal solution is not limited to one point but rather relies on several points simultaneously, and therefore the knowledge of initial feasible points is not required and such points do not influence the final solution. Moreover, a major advantage of genetic algorithms over other stochastic techniques is the availability of several multiobjective techniques as VEGA (Schaffer, 1985), MOGA (Fonseca & Fleming, 1993), NSGA (Srinivas & Deb, 1995), Niche Pareto GA (Horn & Nafpliotis, 1994), and NSGA-II (Deb, Agrawal, Pratap, & Meyarivan, 2000). NSGA-II the most popular multiobjective technique, and it is robust, easy to implement and very fast. Also for genetic algorithms, there are several techniques to handle constraints, such as those proposed by Coello-Coello (2000) and Fujii, Shimoyama, and Oyama (2005). Because of, genetic algorithms offer appropriate techniques to solve problems of a multiobjective and constrained nature. Many studies have applied genetic algorithms to design in chemical engineering (Fraga & Matias, 1996; Gómez-Castro, Segovia-Hernández, Hernández, Gutiérrez-Antonio, & Briones-Ramírez, 2008; Gutiérrez-Antonio & Briones-Ramírez, 2009; Leboireiro & Acevedo, 2004) because

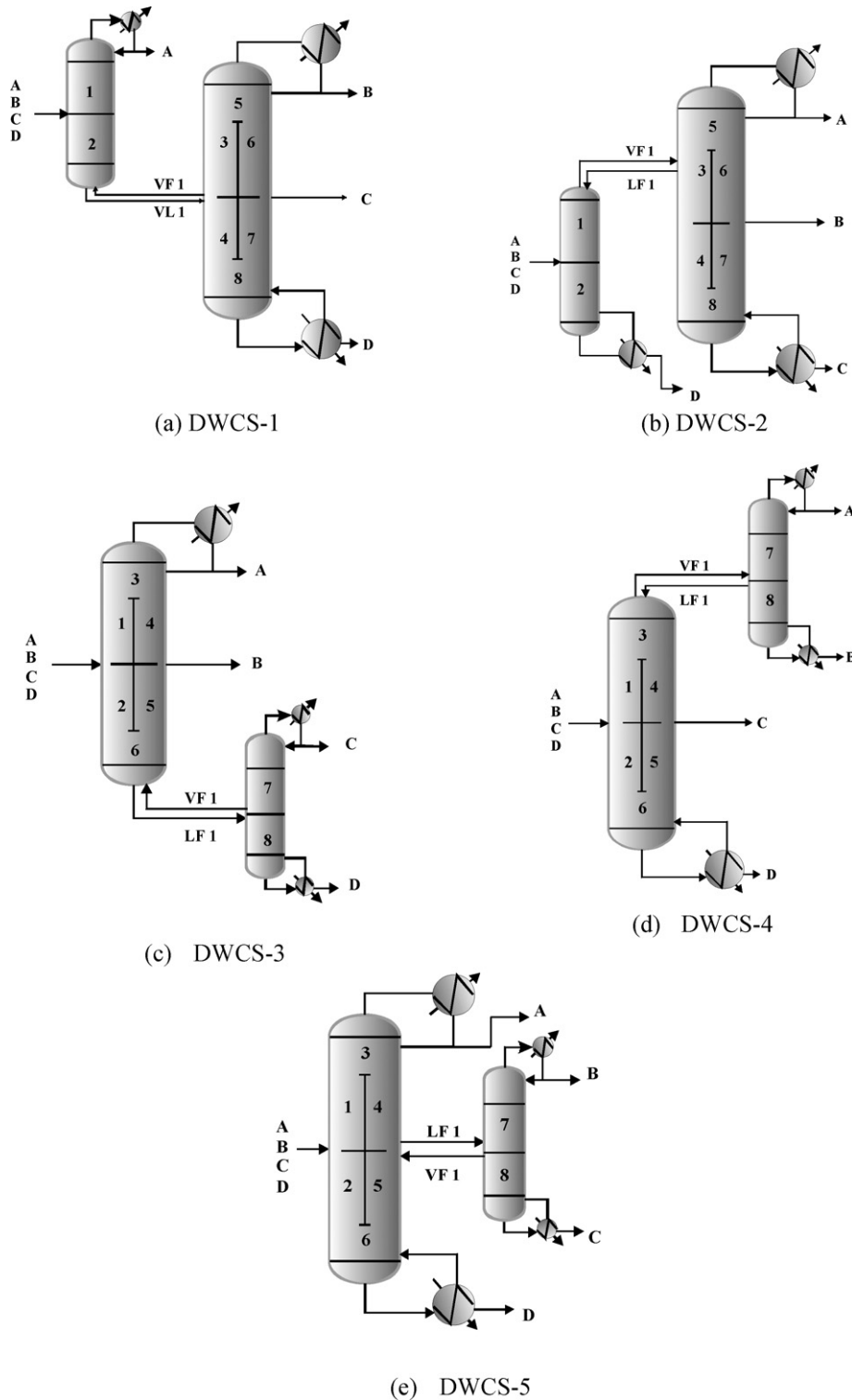


Fig. 1. Intensified distillation systems for quaternary separations.

genetic algorithms are robust and can handle both MESH equations and phase equilibrium calculations with complete models, unlike optimizations made through mathematical programming, in which simplified models for MESH equations and equilibrium phase calculations are used (Caballero & Grossmann, 2001; Kim, 2001, 2005; Pistikopoulos & Proios, 2005).

In this paper, we have taken a subset of five intensified configurations, for quaternary distillations (Fig. 1), of those reported by

Rong and Turunen (2006). We have studied design and optimization using a multiobjective genetic algorithm with restrictions coupled to the Aspen Plus™ process simulator (Gutiérrez-Antonio & Briones-Ramírez, 2009) for the evaluation of the objective function, ensuring that all results obtained are rigorous. Numerical performance of this method has been tested in the design of columns with several mixtures to examine the effect of the relative volatilities of feed mixtures. The study is complemented by a preliminary

control property analysis of the structures obtained with the algorithm. In general, numerical performance shows that this method is robust and suitable for the design of intensified distillation systems for multicomponent separations.

## 2. Formulation of the optimization problem

Optimal design of the new intensified distillation systems implies the determination of 16 variables among continuous and integers, such as total number of stages, location of feed stages, location of the exits and entrances of interconnection flows, reflux ratio, product and interconnection flows. The elevated number of variables along with the enthalpy and phase equilibrium calculations for quaternary mixtures makes this problem mixed-integer non-linear. Optimal design for these sequences means having a structure with as few as possible stages and as small as possible heat duty, but satisfying the purities required.

The optimal design problem can be expressed as:

$$\begin{aligned} \text{Min}(Q_i, N_j) &= f(R, N_k, N_l, F_k, N_F, N_S) \\ \text{subject to} & \\ \bar{y}_m &\geq \bar{x}_m \end{aligned} \quad (1)$$

Where  $R$  is the reflux ratio,  $N_k$  is the stage number of the outlet interconnection flow  $k$  in column  $j$ ,  $N_l$  is the number of stage of the inlet interconnection flow  $l$  in column  $j$ ,  $F_k$  is the value of the interconnection flow  $k$ ,  $N_F$  is the number of stage of the feed stream,  $N_S$  is the number of stage of the side stream,  $\bar{y}_m$  and  $\bar{x}_m$  are the vectors of obtained and required purities for the  $m$  components, respectively.

In order to have optimal designs in sequences DWCS-1 and DWCS-4, we have to minimize four objectives: total number of stages in the three columns and the heat duty of the unique reboiler. Additionally, for sequences DWCS-2, DWCS-3 and DWCS-5 there are five objectives: total number of stages in the three columns plus the heat duty of two reboilers. Also, for the five sequences the required purities for each component must be achieved.

To solve this problem we used the multiobjective genetic algorithm with constraints developed by Gutiérrez-Antonio and Briones-Ramírez (2009). Their code manages constraints with a multiobjective technique, which guides the NSGA-II search (Deb et al., 2000) using the concept of dominance presented by Coello-Coello (2000). Since this code is coupled to the Aspen Plus™ simulator, all optimal designs are obtained considering the calculation of phase equilibrium along with the complete set of MESH equations, using the Radfrac module. For more detailed information about this algorithm and its link to Aspen Plus™, the reader is referred to the original work (Gutiérrez-Antonio & Briones-Ramírez, 2009). It is worth mentioning that, for the optimization of distillation sequences, we also try multiobjective differential evolution (Xue, Sanderson, & Graves, 2003), without carrying out a formal experiment; we have found that, for these kinds of problems, the NSGA-II (Deb et al., 2000) with management restrictions (Coello-Coello, 2000) shows better performance.

Thus, the optimization search includes the manipulation of 16 variables to minimize 4 or 5 objectives, subject to 4 constraints; the number of objectives depends on the sequence that is being optimized, since every structure is optimized independently. Since product flows are manipulated, we consider, as additional constraints, the recoveries of each component (4) of interest to avoid a product flow value decrease. Therefore, we have eight constraints in total.

For all sequences and mixtures, runs were performed with 2000 individuals over 60 generations; these parameters were obtained previously through a tuning process of the genetic algorithm to ensure the convergence in a reduced number of generations, but at the same time avoiding premature convergence. The time

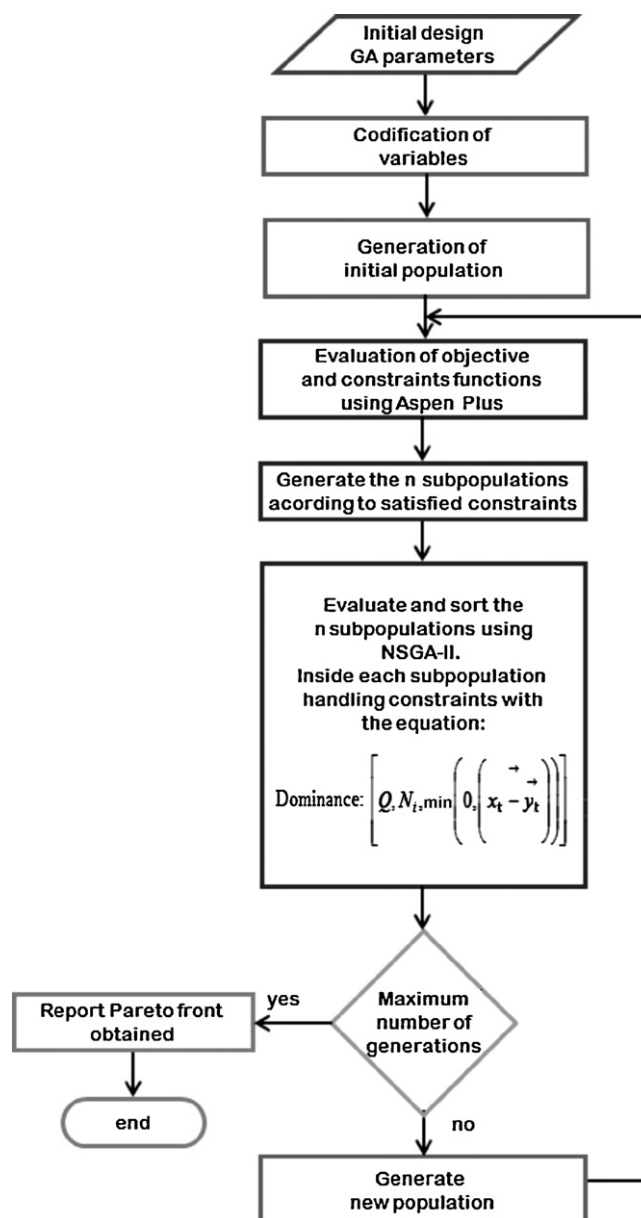


Fig. 2. Block diagram for the genetic algorithm.

employed for the optimization of each sequence is between 8 and 10 h on a Xeon 5410 workstation at 2.33 GHz with 8 GB of RAM. Of the total computational time required, 95% is consumed by the simulations performed in Aspen Plus™, due to the high number of interconnection flows (6) in these sequences; in other words, the most time-consuming activity is the evaluation of objective and constraint functions. As mentioned above, in this study we employed 2000 individuals and 60 generations; this means that, in each generation, 2000 individuals – different combinations of design variables for a single structure – are simulated by Aspen Plus™. Fig. 2 displays a block diagram for the genetic algorithm.

## 3. Case of study

The description of the mixtures and the composition in the feed used in this paper is given in Table 1; the feed flowrate was 45.36 kmol h<sup>-1</sup> as saturated liquid, and the specified purities for

**Table 1**  
Mixtures analyzed and some properties of components.

Component	Feed composition (mol. fraction)	Boiling point (K)	Compressibility factor	Critical temperature (K)	Critical pressure (bar)
<b>Mixture M1</b>					
n-Pentane (A)	0.15	309.22	0.269	469.70	33.65
n-Hexane (B)	0.35	341.88	0.266	506.40	30.30
n-Heptane (C)	0.15	371.61	0.261	539.20	27.40
n-Octane (D)	0.35	398.83	0.258	568.40	24.90
<b>Mixture M2</b>					
2-methyl-butane (A)	0.15	301.0	0.270	433.75	31.99
n-Pentane (B)	0.35	309.22	0.269	469.70	33.65
2-Methyl-pentane (C)	0.15	333.44	0.269	504.33	31.24
n-Hexane (D)	0.35	341.88	0.266	506.40	30.30
<b>Mixture M3</b>					
Benzene (A)	0.15	353.27	0.272	562.61	49.24
Toluene (B)	0.35	439.33	0.263	593.94	40.52
Ethylbenzene (C)	0.15	409.33	0.265	619.72	37.23
o-Xylene (D)	0.35	417.55	0.266	632.22	36.54
<b>Mixture M4</b>					
Methanol (A)	0.15	337.66	0.228	513.16	79.53
1-Propanol (B)	0.35	355.44	0.249	508.33	47.64
1-Pentanol (C)	0.15	411.13	0.272	307.0	40.12
1-Octanol (D)	0.35	468.4	0.310	658.0	34.0

**Table 2**  
Design variables of DWCS-1, M1 mixture.

	Sections 1 and 2	Dividing wall column			
		Sections 3 and 6	Sections 4 and 7	Section 5	Section 8
Number of stages	27	10	18	10	12
Feed stage	12	–	–	–	–
Side stream stage	–	18	–	–	–
Interlinking stages	27/27	5/10	–	–	–
Distillate rate, kmol h <sup>-1</sup>	6.8	–	–	15.96	–
Bottom rate, kmol h <sup>-1</sup>	72.6	–	–	–	6.64
Feed flowrate, kmol h <sup>-1</sup>	45.36	–	–	–	–
Reflux ratio	3.65	2.77	2.77	2.77	2.77
Temperature of distillate, °C	37.3	–	–	70.3	–
Top pressure, atm	1	–	–	1	–
Diameter, m	0.52	0.87	0.87	0.87	0.87
Liquid phase interlinking flow (LF1), kmol h <sup>-1</sup>	72.6	–	–	–	–
Vapor phase interlinking flow (VF1), kmol h <sup>-1</sup>	–	–	34.1	–	–

the product streams were assumed to be 98.7, 98, 98 and 98.6 mole percent for A, B, C and D respectively. The design pressure for each separation was chosen to ensure the use of cooling water in the condensers. Since the feeds M1 and M2 involve a hydrocarbon mixture, the Chao-Seader correlation was used for the prediction of thermodynamic properties. This model is usually recommended for petrochemical plants operating at low or medium pressure (Aspen Plus Manual, 2007; Errico, Tola, & Mascia, 2009). In the case of M3 and M4, the UNIQUAC model was used for calculations.

#### 4. Results

The results are shown in two sections; in the first section, the five thermally coupled distillation sequences are compared in terms of energy consumption, thermodynamic efficiencies ( $\eta$ ; Seader & Henley, 1998), CO<sub>2</sub> emissions (Gadalla, Olujic, Jansens, Jobson, & Smith, 2005) and total annual costs (Turton, Bailie, Whiting, & Shaeiwitz, 2004), obtained by using steady state simulations. The second section presents the theoretical control properties obtained in the SVD analysis (see Appendix A)

**Table 3**  
Design variables of DWCS-2, M1 mixture.

	Sections 1 and 2	Dividing wall column			
		Sections 3 and 6	Sections 4 and 7	Section 5	Section 8
Number of stages	28	10	4	13	9
Feed stage	12	–	–	–	–
Side stream stage	–	16	–	–	–
Interlinking stages	28/28	10/9	–	–	–
Distillate rate, kmol h <sup>-1</sup>	68.1	–	–	6.7	–
Bottom rate, kmol h <sup>-1</sup>	6.75	–	–	–	15.98
Feed flowrate, kmol h <sup>-1</sup>	45.36	–	–	–	–
Reflux ratio	0.38	18.80	18.80	18.80	18.80
Temperature of distillate, °C	85.9	–	–	48.7	–
Top pressure, atm	1	–	–	1	–
Diameter, m	0.78	0.94	0.94	0.94	0.94
Liquid phase interlinking flow (LF1), kmol h <sup>-1</sup>	–	29.5	–	–	–
Vapor phase interlinking flow (VF1), kmol h <sup>-1</sup>	68.1	–	–	–	–

**Table 4**

Design variables of DWCS-3, M1 mixture.

	Sections 7 and 8	Dividing wall column			
		Sections 1 and 4	Sections 2 and 5	Section 3	Section 6
Number of stages	88	7	13	10	16
Feed stage	–	8	–	–	–
Side stream stage	–	17	–	–	–
Interlinking stages	17/18	–	–	–	16/16
Distillate rate, kmol h <sup>-1</sup>	15.88	–	–	6.8	–
Bottom rate, kmol h <sup>-1</sup>	6.62	–	–	–	97.4
Feed flowrate, kmol h <sup>-1</sup>	–	45.36	–	–	–
Reflux ratio	0.81	8.4	8.4	8.4	8.4
Temperature of distillate, °C	100.6	–	–	48.7	–
Top pressure, atm	1	–	–	1	–
Diameter, m	1	0.71	0.71	0.71	0.71
Liquid phase interlinking flow (LF1), kmol h <sup>-1</sup>	–	–	–	–	97.4
Vapor phase interlinking flow (VF1), kmol h <sup>-1</sup>	74.85	–	–	–	–

**Table 5**

Design variables of DWCS-4, M1 mixture.

	Sections 7 and 8	Dividing wall column			
		Sections 1 and 4	Sections 2 and 5	Section 3	Section 6
Number of stages	23	11	9	8	17
Feed stage	–	10	–	–	–
Side stream stage	–	17	–	–	–
Interlinking stages	14/19	–	–	1/1	–
Distillate rate, kmol h <sup>-1</sup>	6.69	–	–	85.2	–
Bottom rate, kmol h <sup>-1</sup>	15.9	–	–	–	6.8
Feed flowrate, kmol h <sup>-1</sup>	–	45.36	–	–	–
Reflux ratio	14.51	0.67	0.67	0.67	0.67
Temperature of distillate, °C	48.6	–	–	–	–
Top pressure, atm	1	–	–	1	–
Diameter, m	0.83	0.8	0.8	0.8	0.8
Liquid phase interlinking flow (LF1), kmol h <sup>-1</sup>	62.6	–	–	–	–
Vapor phase interlinking flow (VF1), kmol h <sup>-1</sup>	–	–	–	85.2	–

**Table 6**

Design variables of DWCS-5, M1 mixture.

	Sections 7 and 8	Dividing wall column			
		Sections 1 and 4	Sections 2 and 5	Section 3	Section 6
Number of stages	28	14	3	9	15
Feed stage	–	15	–	–	–
Side stream stage	–	–	–	–	–
Interlinking stages	13/15	11/15	–	–	–
Distillate rate, kmol h <sup>-1</sup>	16.04	–	–	6.73	–
Bottom rate, kmol h <sup>-1</sup>	15.95	–	–	–	6.6
Feed flowrate, kmol h <sup>-1</sup>	–	45.36	–	–	–
Reflux ratio	1.65	12.32	12.32	12.32	12.32
Temperature of distillate, °C	70.1	–	–	37.3	–
Top pressure, atm	1	–	–	1	–
Diameter, m	0.7	0.83	0.83	0.83	0.83
Liquid phase interlinking flow (LF1), kmol h <sup>-1</sup>	–	44.3	–	–	–
Vapor phase interlinking flow (VF1), kmol h <sup>-1</sup>	12.25	–	–	–	–

derived from open-loop dynamic simulations in Aspen Dynamics.

#### 4.1. Steady state study

The tray arrangements and some parameters for five sequences, after the optimization task, for the M1 case of study are given in Tables 2–6. It can be noted that, for the case M1 (Table 7), the DWCS-1 sequence has the lowest energy requirement and CO<sub>2</sub> emissions, but the DWCS-4 option presents the minimum total annual cost and the highest thermodynamic efficiency. This result is in agreement with the fact that the optimum scheme must be selected in terms of total annual cost, because the same energy requirements in complex distillation sequences can be translated into different costs because of their dependence on the temperatures of the inte-

grated distillation sequence reboilers. The worst option in TAC value is DWCS-3.

In the case of mixture M2 (Table 8), the best option in TAC value is the DWCS-2 arrangement. However, this sequence does not show

**Table 7**Energy consumption, total annual cost, thermodynamic efficiency and CO<sub>2</sub> emissions for M1 mixture.

Sequence	Q (kW)	TAC (USD)	$\eta$ (%)	CO <sub>2</sub> emissions (kg/h)
DWCS-1	741.3	531,287.1	21.4	180.7
DWCS-2	1006.8	553,691.7	19.6	245.4
DWCS-3	743.4	680,975.9	21.8	181.2
DWCS-4	774.8	499,941.4	22.1	193
DWCS-5	1031.9	579,412.7	17.6	251.5

**Table 8**

Energy consumption, total annual cost, thermodynamic efficiency and CO<sub>2</sub> emissions for M2 mixture.

Sequence	Q (kW)	TAC (USD)	η (%)	CO <sub>2</sub> emissions (kg/h)
DWCS-1	2176.4	1,274,254.2	7.9	504.8
DWCS-2	2230.6	1,185,176.7	10.9	489.7
DWCS-3	1887.4	1,354,303.7	11.2	408.1
DWCS-4	1746.7	1,200,221.7	12.9	389.5
DWCS-5	1639.2	1,142,089.4	14.4	359.8

**Table 9**

Energy consumption, total annual cost, thermodynamic efficiency and CO<sub>2</sub> emissions for M3 mixture.

Sequence	Q (kW)	TAC (USD)	η (%)	CO <sub>2</sub> emissions (kg/h)
DWCS-1	1684.0	1,082,299.2	7.6	442.8
DWCS-2	1898.3	1,073,329.4	7.02	481.1
DWCS-3	2174.8	1,018,590.6	5.8	551.2
DWCS-4	1616.9	1,067,605.1	8.95	409.7
DWCS-5	2376.1	1,219,388.7	6.2	604.9

the best values in energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency. When those parameters are analyzed, the best option is DWCS-5 (the second best arrangement in TAC value). In general, for this case, the DWCS-5 option would be the better alternative. Once again, the worst option, in total annual cost, is the DWCS-3 sequence.

Table 9 displays the results for the M3 mixture. The DWCS-3 arrangement shows the minimum value in the TAC calculation,

**Table 10**

Energy consumption, total annual cost, thermodynamic efficiency and CO<sub>2</sub> emissions for M4 mixture.

Sequence	Q (kW)	TAC (USD)	η (%)	CO <sub>2</sub> emissions (kg/h)
DWCS-1	813.7	589,547.1	22.5	243.4
DWCS-2	854.5	434,830.6	24.5	245.8
DWCS-3	706.5	408,346.1	26.5	201.7
DWCS-4	778.3	436,250.0	27.7	222.2
DWCS-5	1102.4	477,045.8	19.1	314.6

and the second worst values in energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency. When those parameters are analyzed, the best option is DWCS-4 (the second best arrangement in TAC value). The worst option in all parameters of study is DWCS-5.

When the M4 mixture is analyzed (Table 10) the best option is seen to be DWCS-3 in TAC, energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency values. The worst arrangement in total annual cost value is DWCS-1, and the DWCS-5 sequence shows the worst values in energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency.

Preliminary heuristic rules can be suggested for this study: when the mixture to separate contains hydrocarbon-like compounds, the best option is presented when component D is purified in a DWC (DWCS-4 or DWCS-5). When the mixture to be separated contains aromatic or alcohol compounds, the best option is presented when component D is purified in a conventional sequence (DWCS-3).

**Table 11**

Transfer function matrix for DWCS-1, M1 mixture.

	R <sub>1</sub>	R <sub>2</sub>	L	Q <sub>3</sub>
X <sub>A</sub>	$\frac{5.0424}{1.02889s+1}$	$\frac{1.4252}{1.96806s+1}$	$\frac{0.1396}{0.5727s+1} + \frac{-0.5596}{(1.19738s+1)(2.02208s+1)}$	$\frac{-4.15}{(1.26872+1)(1.0538s+1)}$
X <sub>B</sub>	$\frac{1.4352}{1.50427s+1} + \frac{-0.1588}{(1.25528s+1)(1.34871s+1)}$	$\frac{3.3276}{0.91092s+1}$	$\frac{0.1404}{0.91908s+1}$	$\frac{-7.5108}{0.689s+1}$
X <sub>C</sub>	$\frac{0.4804}{(2.5735s+1)(0.67629s+1)}$	$\frac{1.0112}{1.56452s+1}$	$\frac{-0.966}{2.54067s+1}$	$\frac{-5.4028}{(1.1211s+1)(1.32429s+1)}$
X <sub>D</sub>	$\frac{-2.9644}{2.5875s+1}$	$\frac{-5.1556}{1.81709s+1}$	$\frac{1.6584}{1.94618s+1}$	$\frac{4.1196}{0.91908s+1}$

**Table 12**

Transfer function matrix for DWCS-2, M1 mixture.

	R <sub>1</sub>	L	Q <sub>2</sub>	Q <sub>3</sub>
X <sub>A</sub>	$\frac{5.0520}{0.86394s+1}$	$\frac{-0.0492}{0.98688s+1} + \frac{0.1256}{0.98719s+1}$	$\frac{-4.4820}{0.85698s+1}$	$\frac{-5.7676}{(1.11534s+1)}$
X <sub>B</sub>	$\frac{-4.9052}{(2.55623s+1)(1.32571s+1)}$	$\frac{0.0108}{0.022425s+1} + \frac{-0.2724}{3.578077s+1}$	$\frac{0.2328}{0.20875s+1} + \frac{-0.2652}{0.88412s+1}$	$\frac{-0.6708}{1.47799s+1}$
X <sub>C</sub>	$\frac{-6.5204}{1.23057s+1} + \frac{0.7520}{2.07769s+1}$	$\frac{1.5992}{2.864068s+1}$	$\frac{1.7676}{0.77887s+1}$	$\frac{0.6236}{0.66842s+1} + \frac{-4.8152}{4.73838s^2+3.36417s+1}$
X <sub>D</sub>	$\frac{-5.3176}{(2.6572s+1)(1.38095s+1)}$	$\frac{1.8328}{(5.64912s+1)(1.121450s+1)}$	$\frac{2.1516}{(3.80345s+1)(0.74503s+1)}$	$\frac{5.5228}{0.82852s+1}$

**Table 13**

Transfer function matrix for DWCS-3, M1 mixture.

	R <sub>1</sub>	L	R <sub>3</sub>	Q <sub>3</sub>
X <sub>A</sub>	$\frac{7.262}{1.12995s+1}$	$\frac{-0.326}{2.29107s+1}$	$\frac{-0.148}{1.21095s+1}$	$\frac{-0.254}{0.73563s+1}$
X <sub>B</sub>	$\frac{2.646}{2.37118s+1}$	$\frac{-1.776}{3.94921s+1}$	$\frac{0.048}{0.3s+1} + \frac{-0.084}{1.93177s+1}$	$\frac{0.038}{0.23s+1} + \frac{-0.112}{2.20s+1}$
X <sub>C</sub>	$\frac{-0.03}{6.05208s+1}$	$\frac{0.008}{15.749s+1}$	$\frac{0.142}{0.57132s+1}$	$\frac{-0.498}{0.910s+1} + \frac{0.050}{1.665s+1}$
X <sub>D</sub>	$\frac{-5.718}{3.34642s+1}$	$\frac{4.164}{5.2965s+1}$	$\frac{-2.442}{4.45108s+1}$	$\frac{24.292}{9.01236s+1}$

**Table 14**  
Transfer function matrix for DWCS-4, M1 mixture.

	$R_1$	$Q_1$	$L$	$Q_3$
$X_A$	$\frac{5.1536}{0.85376s+1}$	$\frac{-0.8552}{(0.11236s+1)(0.95024s+1)}$	$\frac{0.4480}{(0.58s+1)(2.565s+1)}$	$\frac{-9.0768}{1.08468s+1}$
$X_B$	$\frac{-2.4844e^{-1.85s}}{(1.10141s+1)(1.67836s+1)}$	$\frac{0.0464}{0.27565s+1} + \frac{-0.0012}{0.50944s+1}$	$\frac{0.0112e^{-2.58s}}{5.54445s+1} + \frac{-0.004e^{-2.58s}}{14.84692s+1}$	$\frac{-5.8140}{2.91804s+1}$
$X_C$	$\frac{-7.4336e^{-1.6s}}{(1.30557s+1)(1.97507s+1)}$	$\frac{-0.0100}{0.10000s+1} + \frac{0.0308}{1.10000s+1}$	$\frac{-0.2588}{7.13218s+1}$	$\frac{4.1488}{2.30387s+1} + \frac{-6.7828}{(5.37059s+1)(2.95948s+1)}$
$X_D$	$\frac{-15.8532e^{-2.9s}}{(1.58561s+1)(1.26765s+1)}$	$\frac{0.0644}{(3.43330s+1)(0.39026s+1)}$	$\frac{3.9784}{5.27677s+1}$	$\frac{5.3672}{1.084s+1}$

**Table 15**  
Transfer function matrix for DWCS-5, M1 mixture.

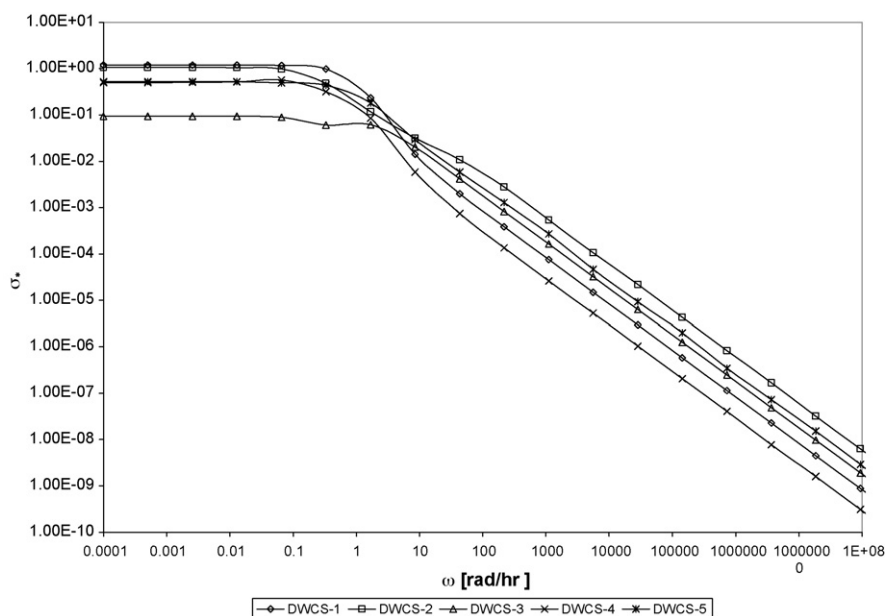
	$R_1$	$R_2$	$Q_2$	$Q_3$
$X_A$	$\frac{3.7344}{0.57048s+1}$	$\frac{0.2820}{(0.3734s+1)(1.48129s+1)}$	$\frac{-2.2192}{(1.57905s+1)(1.13075s+1)}$	$\frac{-8.0284}{0.85582s+1}$
$X_B$	$\frac{-4.47}{(0.43934s+1)(0.72006s+1)}$	$\frac{0.1664e^{-0.02s}}{0.51797s+1} + \frac{-0.04203e^{-0.02s}}{1.19474s+1}$	$\frac{-1.2360}{(0.9494s+1)(1.23023s+1)}$	$\frac{0.69}{0.454s+1} + \frac{-0.2196}{1.11475s+1}$
$X_C$	$\frac{-6.4080}{(0.29525s+1)(0.78261s+1)}$	$\frac{-2.3012}{(0.28746s+1)(0.8679s+1)}$	$\frac{2.6404}{0.88691s+1}$	$\frac{2.0128}{0.97552s+1} + \frac{-0.036}{1.36128s+1}$
$X_D$	$\frac{-7.3780}{(0.17992s+1)(0.93493s+1)}$	$\frac{0.6376}{(3.53440s+1)(1.09887s+1)}$	$\frac{-4.9008e^{-0.80s}}{(2.22361s+1)(1.57877s+1)}$	$\frac{3.7416}{1.367s+1}$

4.2. Control properties study

The SVD technique requires a transfer function matrix (see Eq. (A.1) in Appendix A) around the optimum design of the distillation sequences. For the distillation sequences presented in this study, four controlled variables were considered, product composition A, B, C, D. Similarly, the following manipulated variables were defined: reflux ratios ( $R_j$ ), heat duties supplied to reboilers ( $Q_j$ ) and side stream flowrate ( $L$ ). In this study, step changes in the input variables were implemented and open-loop dynamic responses were registered. The dynamic responses were adjusted to transfer functions and arranged into transfer function matrices. Tables 11–15

present typical transfer function matrices for separation of the M1 mixture.

Figs. 3 and 4 show the Morari resiliency index ( $\sigma^*$ ) and condition number ( $\gamma$ ), in the frequency domain, for all sequences (M1 mixture). A clear trend in the parameters is observed for this separation task: the worst scheme option in TAC value (DWCS-3) shows the highest condition number and the lowest Morari resiliency index at low frequencies. It can be expected that the DWCS-3 option will present the worst closed-loop dynamic behavior for both set point tracking and load rejection, as compared to the other distillation sequences studied. In general, DWCS-3 is a poor option for all parameters studied. For the M1 mixture, the best options for



**Fig. 3.** Morari resiliency index for M1 mixture.

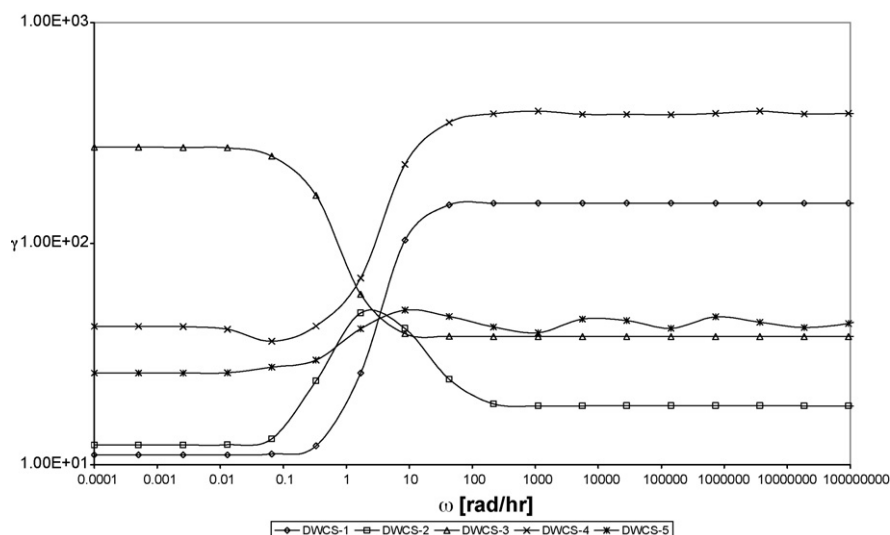


Fig. 4. Condition numbers for M1 mixture.

values of TAC, energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency are the DWCS-1 and DWCS-4 schemes. When they are analyzed as to control properties, the DWCS-1 scheme shows the best control properties (the lowest condition number values and the highest MRI). In the case of DWCS-4, its control property values ( $\sigma^*$  and  $\gamma$ ) are intermediate, between the worse and better schemes. As a result, it can be expected that the DWCS-1 and DWCS-4 sequences will have good closed-loop dynamic behavior. In general, it can be established that the sequence with better TAC, energy consumption, CO<sub>2</sub> emissions and thermodynamic efficiency will show good behavior for both set point tracking and load rejection. Similar results have been obtained for the other cases of study.

## 5. Conclusions

A design methodology for intensified quaternary distillation systems (using dividing wall columns) has been presented. This methodology is based on stochastic optimization techniques, namely genetic algorithms. Five complex distillation systems have been analyzed. The design and optimization methodology used has proven to be an important tool to resolve these kinds of problems, producing results close to the global optimum and with low mathematical effort. Because of the complex nature of the studied arrangements, this rigorous simulation method is absolutely necessary to ensure that the best solution is chosen. Nevertheless, to make the final decision regarding which system is better, an analysis of thermodynamic efficiency, CO<sub>2</sub> emissions and control properties should be carried out. Preliminary heuristic rules can be suggested for this study: when the mixture to separate contains hydrocarbon compounds, the best option is presented when component D is purified in a DWC (DWCS-4 or DWCS-5). When the mixture to be separated contains aromatic or alcohol compounds, the best option is presented when component D is purified in a conventional sequence (DWCS-3). The best options in energy savings, TAC,  $\eta$  and CO<sub>2</sub> emissions values also show, in general, the best control properties. As a result, good dynamic closed-loop performance can be expected for these types of intensified distillation sequences simultaneously with energy savings, total annual costs, thermodynamic properties and greenhouse gas emissions parameters.

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## Appendix A. Singular value decomposition

One definition of SVD is:

$$G = V\Sigma W^H \quad (\text{A.1})$$

Here,  $G$  is the matrix target for SVD analysis and  $\Sigma$  is a diagonal matrix which consists of the singular values of  $G$ . The singular values of the open-loop frequency function matrix of a process at a given frequency are the gains of the process at this frequency in the directions of the corresponding input singular vectors (as the input singular vectors form a basis in the input space, the gain can be calculated in every direction). These gains play an important role when performing controllability analysis of a process, and for a complex analysis they must be evaluated at various frequencies. The Morari Resiliency Index (MRI) is the smallest singular value ( $\sigma^*$ ) of the process open-loop frequency function matrix. The larger its value, the more controllable the process is. If it is zero, this means that there is an input direction where the gain is zero and the matrix is not invertible. Condition number ( $\gamma$ ) is the ratio of the largest and smallest singular values of the process open-loop frequency function matrix. If it is large, then the matrix has strong directionality, which means that the gains vary strongly depending on input directions. Such a matrix is said to be ill conditioned. A large  $\gamma$  means that the system is sensitive to input and model uncertainty and therefore the process is less controllable. Systems with higher  $\sigma^*$  values and lower  $\gamma$  are expected to show the best dynamic performance under feedback control (Gabor & Mizsey, 2008).

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