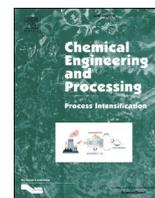




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A Short Review of Dividing Wall Distillation Column as an Application of Process Intensification: Geographical Development and the Pioneering Contribution of Prof. Arturo Jimenez in Latin America

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ABSTRACT

In different geographical areas, various authors have distinguished themselves by substantial contributions to the area of process intensification, and in particular to the study of the dividing wall column (DWC). In the case of Mexico and Latin America, Professor Jimenez and his research group have been pioneers in the study of this complex distillation system. The contributions made by Prof. Jimenez and his doctoral students have been relevant regarding the state of the art of the dividing wall distillation column, since they have extended the studies to design, optimization and control of DWC not only for ternary separations but also for multicomponent separations, extractive, reactive and azeotropic cases in a wide variety of (bio) applications. These contributions have been made in the areas of simulation fields and experimental research, conducting to the implementation of an experimental dividing wall distillation column, is the first one reported in Latin America. This short review aims to show the contribution that Professor Jimenez has made in the study of the DWC, as well as to show the impact that Professor Jimenez has had on the national and international level with his work, particularly in the area of new knowledge in the continuous development of DWCs.

1. Introduction

The XXI century has been markedly characterized by increased environmental awareness and pressure from legislators to society to improve energy efficiency by adopting 'greener technologies'. In this context, the need for the chemical industry to develop processes which are more sustainable or eco-efficient has never been so vital. The successful delivery of green, sustainable chemical technologies at industrial scale will inevitably require the development of innovative processing and engineering technologies that can transform industrial processes in a fundamental and radical fashion. Process intensification (PI) can provide such sought-after innovation of equipment design and processing to enhance process efficiency. PI aims to make dramatic reductions in plant volume, ideally between 100- and 1000-fold, by replacing the traditional unit operations with a novel, usually compact designs, often by combining two or more traditional operations in one

hybrid unit [1]. The PI concept was first established at Imperial Chemical Industries during the late 1970s when the primary goal was to reduce the capital cost of a production system. Although cost reduction was the original target, it quickly became apparent that there were other important benefits to be gained from PI, particularly in respect of improved intrinsic safety and reduced environmental impact and energy consumption, as it will be discussed later in this section. Over the last two decades, the definition of PI has thus evolved from the simplistic statement of 'the physical miniaturization of process equipment while retaining throughput and performance' to the complex definition provide recently by Tian et al. [2]. This definition refers to the summarized activities that result in intensified processes, including the combination of multiple process tasks or equipments into a single unit (e.g., membrane reactors, reactive distillations), the miniaturization of process equipment (e.g., microreactors), the operation of equipment in a periodic manner (e.g., simulated moving bed, pressure adsorption swing),

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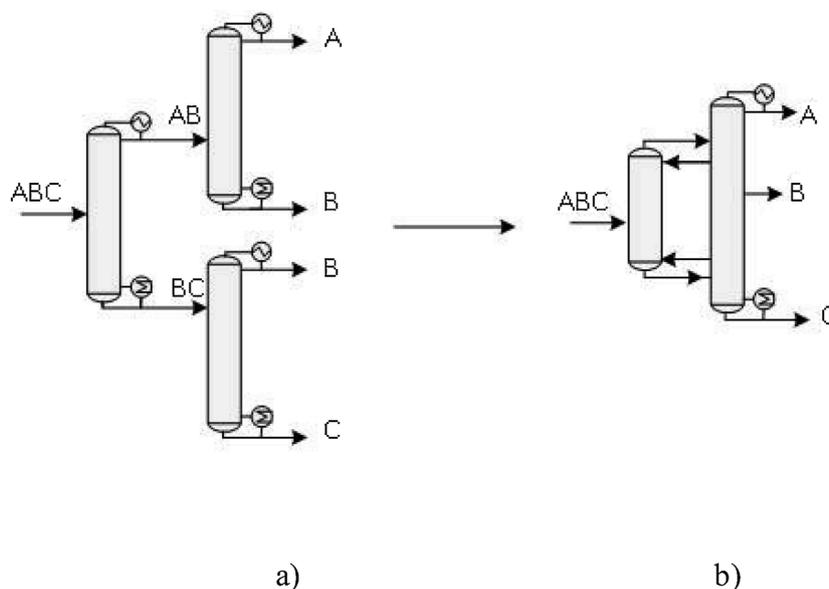


Fig. 1. Conventional sloppy scheme (a) and a Fully thermally coupled Scheme, Peltyuk, (b).

and tight process integration (e.g., dividing wall distillation). Another well accepted PI definition is that proposed by Stankiewicz and Moulijn [3]. They mentioned that PI promotes the development of novel technology and techniques that, compared to conventional techniques, are expected to improve manufacturing and processes. Additionally, a decrease in equipment size, energy consumption, and waste production is expected, which would be reflected in cheaper and more sustainable technologies.

Judging from the growth of research interest, it is clear that PI is a promising field that can enable a paradigm shift to the process industry, offering novel processing methods and equipment to achieve higher efficiency and safer operation [3–5].

Distillation is widely used to separate fluid mixtures in chemical and allied industries. It is an energy-intensive process, which shares approximately 40% of the total energy consumed in process industries. Surprisingly, distillation offers a low thermodynamic efficiency of 5–10% [6]. To solve these problems, heat integration came forward as an effective technique that aims to improve thermodynamic efficiency via a thermal coupling. There are typically two groups of heat integration: internal and external heat integration. The former type includes heat integrated distillation column (HIDiC) and dividing wall column (DWC), and the latter one includes vapor recompression, bottom flashing, closed-cycle compression and absorption heat pump [7]. This work is concerned with the DWC. The DWC is built in a single shell divided by a wall and is capable of separating mixtures of three or more components into high purity products. Compared to conventional columns, a DWC requires much less energy, capital cost, and space. This makes DWCs correspond to the present-day idea of sustainable process technology [7]. Several studies have been conducted on the design, control, and optimization of DWCs in the past three decades as reported by El-Gendy et al. [8]. These authors show the growing interest in this configuration in recent years, based on data from Scopus provided by Elsevier B.V.

The term of DWC is defined as dividing wall column because the middle part of the column is split into two parts by a wall. Feed, typically containing three or more components (A,B,C), is pumped into one side of the column facing the wall. Deflected by the wall, the lightest component A flows upward and exits the column as top distillate, while the heaviest component C drops down and is withdrawn from the bottom of the column. The intermediate boiling component B is initially entrained up and down with both streams, but the fluid that goes upward subsequently separates and falls down on the opposite side of the wall. Similarly, the amount of B that goes toward the bottom separates and

flows up to the back side of the wall, where the entire B product is recovered by a side draw stream.

Consider a three-product separation as indicated in Fig. 1a in which the lightest and heaviest components are chosen to be the key separation in the first column. Two further columns are required to produce pure products. This arrangement is known as distributed distillation or sloppy distillation. The distillation sequence provides parallel flow paths for the separation of a product. At first sight, the arrangement in Fig. 1a seems to be inefficient in the use of equipment in that it requires three columns instead of two, with the bottoms and overheads of the second and third columns, both producing pure B. However, it can be a useful arrangement in some circumstances. In a new design, the three columns can, in principle, be operated at different pressures. Also, the distribution of the middle Product B between the second and third columns is an additional degree of freedom in the design. The additional freedom to vary the pressures and the distribution of the middle product gives significant extra freedom to vary the loads and levels at which the heat is added to or rejected from the distillation. This might mean that the reboilers and condensers can be matched more cost-effectively against utilities, or heat integrated more effectively. If the second and third columns in Fig. 1a are operated at the same pressure, then the second and third columns could simply be connected and the middle product taken as a sidestream as shown in Fig. 1b. The arrangement in Fig. 1b is known as a prefractionator arrangement. Note that the first column in Fig. 1b, the prefractionator reduce the overall energy consumption. Comparing the prefractionator arrangement in Fig. 1b with the conventional direct and indirect sequences, the prefractionator arrangement typically requires 20 to 30% less energy than conventional arrangements for the same separation duty. The reason for this difference is rooted in the fact that the distributed distillation and prefractionator arrangements are fundamentally thermodynamically more efficient than a simple arrangement. The cause of the higher efficiency in DWC over a conventional arrangement is that the prefractionator (see Fig. 1b) requires significantly less energy than a conventional separation train. This arrangement avoids the remixing of internal streams, which in a series of two consecutive columns shows a peak in the concentration of the middle component. Additionally, the advantage of considering a prefractionator is the better distribution in the middle component, allowing greater freedom to match the feed composition with a tray in the column to further reduce mixing losses at the feed tray. This mixing or remixing that happens in a conventional arrangement is the inevitable source of thermodynamic inefficiency [9].

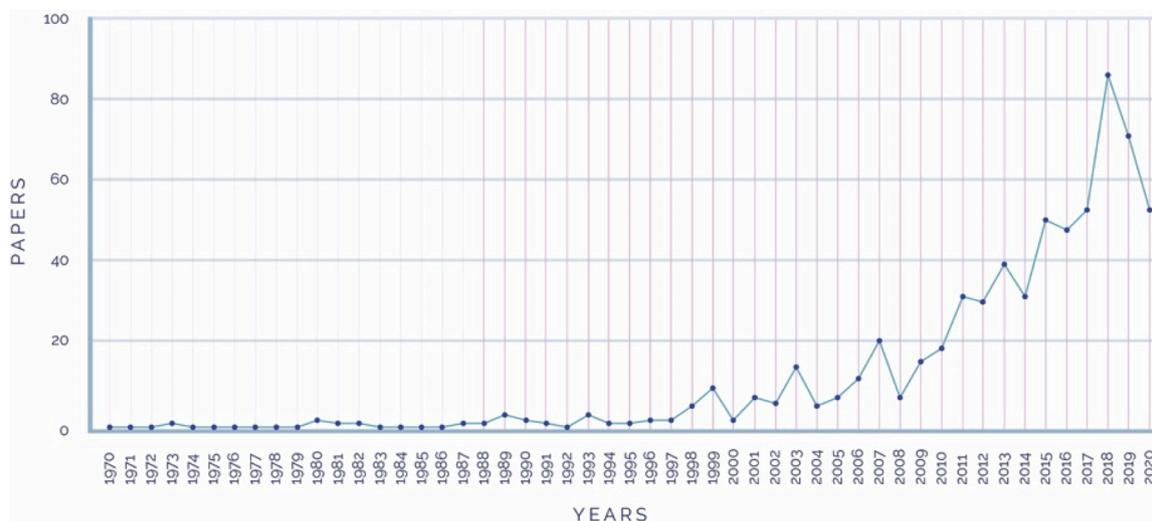


Fig. 2. Research papers related to Petlyuk column or dividing wall column according to a search conducted in the Scopus-Elsevier data base (September 16, 2020).

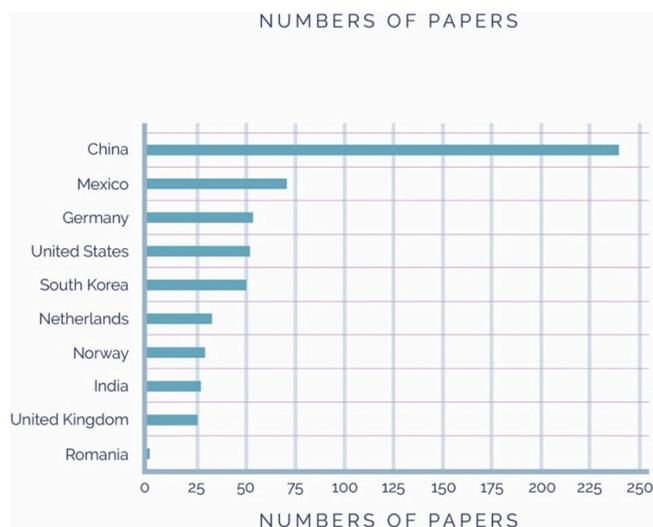


Fig. 3. Research papers published by countries, regarding the topics of Petlyuk column or dividing wall column according to a search conducted in the Scopus-Elsevier data base (September 16, 2020).

A search of the phrases “Petlyuk column” or “dividing wall column” in the Scopus-Elsevier data base displays 638 research papers and they are distributed in time according to Fig. 2. It is important to mention that the number of documents related to these topics has increased dramatically in the last 20 years. This could be due to the fact that these complex distillation columns were important in areas such as process intensification, process control, energy savings, biofuels, etc.

Regarding where these papers are published Fig. 3 illustrates that China has published 234 papers. Mexico has published 74 papers, ranking second internationally and ranking first in Latin America concerning the generation of knowledge in reference to DWC. It is expected that the number of papers will continue to increase because this technology has been adopted by industry.

In regards to DWCs, the publication of works can be noticed in four major geographic regions: the United States of America, Europe, China and Latin America.

China has had a considerable increase in recent years in relation to the other regions. Mexico holds the second place in the world for scientific contributions regarding the DWC. In this sense, the role of Professor Jimenez has been fundamental because he is considered the

pioneer in the scientific production of this particular subject in Mexico. Considering the publication dates, the knowledge generated in Professor Jimenez’s research group has served as a fundamental pillar in the development of DWC research throughout the world.

Thus, this review aims to show the relevant contributions that have been generated in the world that address DWCs to date, and Professor Jimenez’s contributions in regard to the continuous research of DWC.

In the following sections, the authors will examine relevant work in the area of synthesis, design, control and optimization of processes involving the intensification of processes applied in DWCs.

2. Dividing wall column approaches throughout the world

As mentioned, all of the DWC contributions can be separated according to the geographical regions or countries involved in the overview of the publications. The next section will begin with the USA and will finalize with Latin America (without considering Mexico). Once this has been done, we will highlight various research projects developed by Professor Jimenez and his research group, and/or by former students of Professor Jimenez. All these works were mainly carried out in Mexico and for that reason we have decided to present this country separately.

2.1. The United States of America

In the United States of America, as in other parts of the world, the research, development, and physical implementation of dividing wall columns has seen increasing interest in recent decades. In 1986, Chavez et al. [10] modeled the nonlinear equations, in COFRAC, to simulate a Petlyuk distillation column. Their results showed the effects related to the energy saving capacities of Petlyuk-type columns. Additionally, the column’s steady state multiplicities were observed. In another research, Carlberg and Westerberg [11] implemented the Underwood set of equations to design a Petlyuk-type column that works at minimum reflux. Their work was limited to a mixture of three components.

In a later research, Agrawal and Fidkowski [12] analyzed ternary systems to determine if the composition in the feed and/or the volatility of the components had an effect in Petlyuk-type systems. Their case study results demonstrated that in a relatively limited range the Petlyuk-type scheme had a higher thermodynamic efficiency.

Caballero and Grossmann [13] created a disjunctive programming model for the optimal synthesis of distillation columns. Using previous work by Calberg and Westerberg (1989), a modified version of the logic-based outer approximation algorithm was used to solve the resulting model.

Later, Slade et al. [14] presented their work to produce a stream of xylene from reformed in an aromatics plant, by changing conventional distillation columns to a DWC type configuration, and improving product purity and energy savings. Up to this point in history (2009), according to Ling and Luyben, there were approximately 40 reports referring to Peltyuk or DWC type columns, and the control properties had been little explored. In research proposed by Ling and Luyben [15], they presented a control structure based on controlling the compositions of the heaviest components in the prefractionator for disturbances in the composition of the hydrocarbon feed. As a result of this, they showed that a DWC-type scheme improves control properties compared to a conventional distillation train designed with the same separation objective.

The dynamic models of the DWC have also been examined and Joglekar et al. [16] presented a dynamic model for a DWC column to separate benzene, toluene and o-xylene. To determine the optimal operation of the column, they used genetic algorithms modeled in Matlab.

In an optimization framework, Kim and Linninger [17] optimized complex DWC schemes to separate quaternary mixtures. A design-optimization method based on inverse design methods and temperature collocation substantially reduces the problem dimension. As a complementary method, a hybrid algorithm was used initially based on genetic algorithms and finally a deterministic search. As a result of their work, it was concluded that this design-optimization method allows them to generate designs that can save up to 70% of energy compared to conventional separation schemes.

Various azeotropic mixtures were analyzed by Zou et al. [18] through MINLP programming. In their research, they presented the improved state-space (SS) superstructure incorporating all basic mass and heat transfer elements to capture all configurations. The optimal designs presented significant savings that were measured by the total annual cost (TAC).

Although liquid/vapor thermal couplings are a fundamental part of DWC column design, Madenoor-Ramapriya et al. [19] explored various alternatives in which the vapor stream can be replaced by a liquid stream in a thermodynamically equivalent manner. With this in mind, the synthesis work is modified and produces different alternatives to separate quaternary mixtures. As a result of their synthesis work, initial indications are presented of an improvement in the dynamics of this type of alternatives compared to traditional thermal couplings. Later, the same research group, Ramapriya et al. (2016), examined the simplification of this methodology which was reduced to four steps, as well as the generation of a DWC considering thermal couplings and liquid streams as a substitute for thermal couplings.

In an effort to reduce the costs associated with DWC, Long et al. [20] presented an iterative optimization based on the reduction of the design space. Subsequently, through a sensitivity analysis, optimal design parameters were obtained. As case of study, a mixture of hydrocarbons was used, obtaining as a result, improvements in the total annual cost compared to a conventional scheme. On the other hand, Pattison et al. [21] proposed a new approach to model DWCs; they represented DWCs as networks of pseudo-transient (differential-algebraic) subunit models. The authors produced a reactive column for the production of dimethyl ether, obtaining 14% of energetic savings in comparison with the traditional process route.

In the same framework of reactive DWC, Li et al. [22] introduced a rigorous modeling of a reactive DWC for the production of methyl acetate by hydrolysis. Using a rigorous model and sensitivity analysis, energy savings of about 20% were obtained compared to the conventional process. Subsequently, a control study was carried out using a proportional integral controller. Utilizing a temperature control strategy, a reasonable performance was obtained to maintain the purities of the products of interest in the desired purity. On the other hand, Blevins et al. [23], used model predictive control techniques. With the implementation of this model, control parameters could be estimated when

measurements or samples taken in a laboratory column failed. In their work, the operation of the controller is detailed and the challenges in the application of this type of controllers in pilot plant level columns are discussed.

In another interesting work on DWC synthesis, Madenoor et al. [24] (2018a) implemented an easy-to-use step-wise procedure to synthesize an initial-dividing wall column (i-DWC) from any given n-component basic distillation column sequence or its thermally coupled derivative. This procedure is dependent on the nature of the distillation column to be converted to a DWC. Their work significantly expands the search space of useful DWCs to separate any given multicomponent mixture. In a second part of this work, Madenoor et al. [25] presented a simple rule which enables exhaustive enumeration DWCs corresponding to any given thermally coupled distillation configuration. With the successive application of the rule, every partition in a DWC can be extended all the way to the top and/or to the bottom of a column without losing thermodynamic equivalence to the original thermally coupled configuration.

DWCs have also been combined with various techniques to improve energy efficiency. Yang et al. [26] put forth a reactive DWC proposal for the production of diethyl carbonate. Its reactive DWC proposal is assisted by vapor recompression techniques. As a result, it shows a reduction of 20.52% in the total annual cost, as well as a reduction of 33.74% in CO₂ emissions compared to the conventional scheme.

In addition, DWCs have been used to handle trace components in purification operations. Donahue et al. [27] presented an experimental study of a DWC to control the presence of trace components in feed streams. In their work, conventional controller design tools are used to devise multiloop control structures that manage the concentration of a trace feed impurity in the product streams. Through the use of temperature controllers, it is shown that impurities can be relatively well controlled in the operation of a DWC. Additionally, Donahue et al. (2019b) experimentally validated a rigorous model scaled to the size of an industrial column. The model considered heat transfer to analyze the impact of thermal couplings on energy consumption of a DWC. This work shows how a general method of how DWC design can be rigorously approached.

2.2. Europe

Even though its concept was established some 60 years ago, in 1985, BASF put the first industrial DWC into operation, it took more than 10 years after first application at BASF before other companies have realized and consequently experienced that making adequate provisions for separations of three component feeds into pure products within one shell, where appropriate, enables saving of both operating and investment costs. In the following 20 years, around 80% of the installed industrial columns were operated by BASF worldwide. BASF also started up the first 4 products DWC [28–29]. Savings in both energy consumption and fixed investment can be accomplished through the implementation of such a separation scheme. Theoretical studies have shown that DWC can save up to 30% in energy costs compared to conventional schemes [30–32]. Such results have promoted the development of more formal design methods [33–36].

In fact, using DWDCs can save up to 30% in the capital invested and up to 40% in energy costs [37,38]. Moreover, retrofitting conventional column systems into DWC is also a feasible option [39]. However, it should be noted that using DWC requires a match between the operating conditions of the two standalone columns, and due to its design limitations, the main weakness of DWC is its inflexibility to changes in the nature of the feed [40]. Several other successful examples of use of DWC can be found among reactive separations that combine reaction and separation steps in a single unit, such as reactive distillation [41–43].

To promote a stronger potential for its industrial implementation, a proper understanding of the operation and control properties of the DWC was needed to complement the energy savings results. Evidently,

the expectation that the dynamic properties of a complex distillation column may cause control difficulties compared to the rather well-known behavior of the conventional direct and indirect sequences for the separation of ternary mixtures may be one of the factors that had contributed to their lack of industrial implementation. Several efforts, in the 90 s of the XX century had been reported towards the understanding of the dynamic properties of this configuration [44–46].

In the last 20 years there have been interesting contributions that have allowed a better understanding of the design, operability and applications of the DWC in Europe.

Ranger et al. [47] conducted research on an a posteriori method for the multi-objective optimization of DWCs. Optimizing the total stage number, the reboiler duty and the product purities simultaneously results in a high-dimensional solution space. The resulting visualization problem can be solved with self-organizing patch plots. With these plots, a filtration of the results can be performed several times in different scenarios to determine the best suited operating point. This means that optimization-based design is possible without additional calculations.

Egger and Fieg (2019) developed a newly developed rate-based reactive DWC model for packed columns. Their detailed research demonstrates the influence of separation modeling. Comprehensive experimental reactive DWC data are used for the reference system of the butyl acetate transesterification with hexanol and employed for a model validation. The validation results for the NEQ model are compared to the well-known equilibrium stage approach. Following the same line, the influence of azeotropic phase equilibrium on the energy saving potential of the reactive DWC has been analyzed systematically by Harding and Fieg [48]. Their work investigates the influence of non-ideal reaction system properties, focusing on azeotropic phase equilibrium. A comprehensive process is developed with the aim to understand how the non-ideal separation properties affect the energy saving potential of reactive DWC. The results are combined in short heuristics, which allow the process engineer a quick and easy evaluation of the energy saving potential of reactive DWC for the investigated reaction systems during process synthesis.

In an another valuable paper carried out in Europe, Waltermann et al. [49] proposed an optimization-based approach for the design of such complex and integrated distillation processes, enabling a case-specific economic assessment and benchmarking. The application is demonstrated for different case studies, including the separation of an azeotropic quaternary system in a multi-dividing wall column.

Lukac et al. [50] reported results of a simulation study that could bring some light in this respect. They prove that a temperature-based control structure, in conjunction with tight control of temperature profiles in prefractionation section in a DWC, as well as in the product draw regions of the column, is capable of restoring the operation from typical disturbances in feed quality and quantity. Combining temperature-based control with two composition controllers enables the column to be operated within finer margins, resulting in minimal over-purification and overall energy requirement.

A key challenge in DWCs is to ensure an energy efficient operation in case of disturbances. Egger and Fieg [51] aimed at understanding the underlying dependencies of the most important variables in their study: the liquid split, the energy demand and the component distribution, regarding energy efficient control. Based on their findings a straightforward decentralized control structure is presented and analysed. The structure utilizes the liquid split as a manipulated variable in order to control the component composition on the top stage of the prefractionator. The evaluation of the structure regarding energy efficient process control is carried out by comparison to a structure with fixed liquid split.

The production of methyl decanoate through esterification of decanoic acid with methanol by reactive distillation is operationally challenging and energy-intensive due to the complicated behaviour of the reaction system and the difficulty of retaining the reactants together in the reaction region. Methanol, being the lightest component in the

mixture, can separate itself from the reactant as the distillation proceeds which will cause a reduction in the conversion utilizing either a batch or continuous distillation process. Aiming to overcome this type of the potential problem, novel integrated divided-wall batch reactive distillation configuration with recycling from the distillate tank was researched by Aqar et al. [52]. This study has clearly demonstrated that the integrated divided-wall batch reactive distillation column is superior to the traditional conventional batch distillation and both the divided-wall, and split reflux divided-wall batch reactive distillation configurations.

The Lonza AG has successfully developed and implemented an extractive dividing wall column. The entire equipment design was exclusively carried out based upon simulations without carrying out time-consuming pilot or mini plant experiments. This approach considerably shortens the time-to-market cycle and reduces the development costs to a large extent. Despite their great potential to notably reduce costs, only a limited number of extractive dividing wall columns are being operated in the chemical industry and published data is scarce. The paper by Staak and Grützner [53] aimed to close this gap.

The control of a novel heat-pump-assisted extractive distillation process taking place in a DWC was proposed for bioethanol dehydration by Patrascu et al. [54]. This integrated design combines three distillation columns into a single unit that allows over 40% energy savings. This work presents the challenges related to process dynamics and control of this highly integrated system. After showing the control difficulties associated with the original design owing to thermal unbalance, an efficient control structure is proposed which introduces a by-pass and an additional external duty stream to the side reboiler. Two quality control loops ensure product purities when the system is affected by feed flowrate and composition disturbances.

In general, the use of the DWC is recommended for multicomponent liquid mixtures which are separated into at least three fractions with high purity requirements. Furthermore, the DWC is especially favorable for the separation of small quantities of light and heavy boilers from the main middle-boiling product. Cameretti et al. [55] proposed a new configuration of a DWC, which is most effective for the separation of a feed containing low concentrations of middle-boiler (about 1% or less) into a pure distillate and a pure bottoms fraction, while the impurity is concentrated in the side-stream. Product loss in the side-stream is a major cost-driver. In the proposed DWC-configuration, product loss can be cut in half compared to a single side-stream column. Experiments at pilot scale have confirmed the simulation results, proving a new opportunity to profit from the vertical wall inside the distillation column.

Errico et al. [56] introduced a set of new alternative distillation configurations to the four-component DWC. A hydrocarbon and an alcohol mixture are considered to prove their applicability. The total annual cost, the thermodynamic efficiency and the carbon dioxide emissions are considered as comparison indexes between the Petlyuk and the alternative configurations. The results obtained proved that for these cases, at least one of the alternative configurations showed a better or at least the same performance of the DWC arrangement. For this reason, the new configurations are worthy of consideration when four-component mixtures are considered.

Dejanovic et al. [57] addressed the technical feasibility related to aspects of multi-partition wall alternatives for a four-product DWC, which, although highly beneficial, have not been yet attempted in industrial practice. Utilizing an industrially relevant aromatics processing plant case as a basis for design and evaluation of cost-effectiveness of alternative configurations. This work focuses on the hydraulic design and dimensioning of a minimum energy configuration with two overhead product streams. In this research, related issues of DWC technology are discussed and these issues are helpful to distinguish what makes sense and what does not make sense when dealing with practical implementation of multi-partition wall configurations.

Reactive separation technologies were proposed by Kiss and Ignat [58] and Ignat and Kiss [59] for fatty acid methyl esters production,

providing significant benefits, such as minimal capital and operating cost savings. One approach is to use a reactive DWC for the biodiesel production process. However, since the reactive DWC is designed for a quaternary reactive system – two reactants (one in excess) and two products –, more difficulties concerning the process control may be expected considering the high degree of integration of the process. This study is among the first to tackle the optimal design, dynamics and control of such an integrated unit and it proposes an efficient control structure for a biodiesel process based on reactive DWC technology. Aspen Plus and Aspen Dynamics were used as computer aided process engineering tools to perform the rigorous steady-state and dynamic simulations, as well as the optimization of the new reactive DWC based biodiesel process.

A great deal of research is focused on the design and optimization of the DWC and its applications are still reduced due to distrust of its controllability and robustness. Previous studies have examined the decentralized control of DWC, but still few studies deal with how the model predictive control is applied to DWC. Rodríguez-Hernández and Chinea-Herranz [60] presented a decentralized control of both a DWC along with its equivalent MPC scheme, and both approaches are compared. Instead of building a rigorous model or performing the step test to an existing plant, the MPC model is obtained by identification of a rigorous simulation. Their proposal demonstrated to represent adequately the DWC column behavior. This approach might be convenient if plant testing is not available.

The industrial production of anhydrous bioethanol requires energy demanding distillation steps to overcome the azeotropic behaviour of ethanol-water mixture. In 2012, Kiss's research group [61,62] propose novel distillation technologies for enhanced bioethanol dehydration, by extending the use of DWCs to energy efficient azeotropic and extractive distillation. The results of the rigorous Aspen Plus simulations show that energy savings of 10-20% are possible for the novel process intensification alternative based on DWC.

Regarding DWC, Rewagad and Kiss [61] illustrate the advanced control strategy based on MPC to perform dynamic optimization. The dynamic model of the DWC used in this study is not a reduced one, but a full-size nonlinear model that is representative of industrial applications. The quality of the linearized model used for the predictions inside MPC is derived from and tested against the non linear model. The variables are selected to achieve the aim of regulatory and inventory control in the column, and at the same time minimizing the energy requirements in a very practical way. The optimal energy control is based on a simple strategy to control the heavy component composition at the top of the prefractionator side of the DWC by manipulating the liquid split. The performance of the MPC is evaluated against a conventional PID control structure which was previously reported to be the best performing in the operating a DWC.

Rong [63] showed that the DWCs can be systematically generated from the conventional simple column configurations. Because the simple column sequences with sharp splits are simple and widely studied conventional schemes for multicomponent distillation, the purpose of this research is to formulate a procedure for systematic synthesis of DWCs for such simple conventional schemes. A four-step procedure is formulated which systematically generates all the possible DWCs from the simple column sequences. First, the subspace of the original thermally coupled configurations corresponding to the simple column configurations is generated. Then, the subspace of the thermodynamically equivalent structures corresponding to the original thermally coupled configurations is produced. Finally, the subspace of the DWCs corresponding to the thermodynamically equivalent structures is achieved.

A lack of research still exists when dealing with the start-up of dividing-wall columns, which is inherently a strongly nonlinear process. Here, for the first time the start-up of dividing-wall columns was explored by Niggemann et al. [64], where the starting point is an empty column at ambient conditions. A model is presented which is capable of predicting the dynamic discrete-continuous changes which are

characteristic of DWCs. The proposed process model takes into account the heat transfer across the dividing wall, as well as the vapor distribution below the dividing wall. The degree of accuracy of the model is clearly determined by comparing different simplifications, e.g., a constant vapor distribution ratio equal to the steady-state value. The rigorous process model and the obtained simulation results presented in this study provide a promising basis for developing and applying optimal start-up policies for DWCs.

In review papers, Dejanovic et al. [65] and Aspiron and Kaibel [66] provide a complete overview of the work carried out so far on the research and implementation of DWCs, from early ideas of thermal coupling of distillation columns to practical issues that needed to be solved for their successful implementation.

A thermodynamically equivalent structure (TES) is the distinct feature of a thermally coupled configuration. From 2002 to 2008 Rong's research group [63] presented the synthesis and optimal design of thermodynamically equivalent thermally coupled systems and DWCs for multicomponent distillations. First, the original thermally coupled configurations (OTCs) for traditional distillation configurations with sharp splits are generated. Then, generation of all of the possible TESs for the OTCs is presented. Two rules are developed for synthesizing the possible TESs for any OTC involving both sharp and sloppy splits. Heuristics and a simple procedure have been approached to find an optimal TES.

2.3. China

In the particular case of China, the publication of works referring to DWC schemes began in even more recent years than in other regions. However, the growing interest in this country is demonstrated in the large number of papers that have been published in recent decades, placing China above other regions. Many of the works reported in China refer to particular case studies. For example, Lan-Yi et al. [67] proposed the use of an azeotropic DWC for ethanol dehydration. In their work, it was possible to detect the optimal values of the design variables to guarantee a minimum energy consumption. As a result, they obtained an energy saving of 42.17% compared to the conventional distillation options.

Various tools have been used to aid the understanding of DWCs. For example, Wang et al. [68] presented a computational analysis of fluid dynamics (CFD) to predict the hydraulics of a sieve type stage in a DWC. They reported the presence of two backflow regions in the sieve stage.

The control properties of DWCs have also been explored. In particular, Xia et al. [69], in a control study of an extractive DWC, reported a control strategy considering four composition controllers and an adjustable vapor split ratio α_V . Additionally, a QR / F control strategy was used. Both strategies showed the ability to handle disturbances.

Consecutively, Wang et al. [70–72] presented three works where a DWC was used for the purification of aniline, nitrobenzene, and chlorobenzene, respectively. In their articles, the benefits of using DWCs are highlighted, evidenced by energy savings. Another interesting application that showed the benefits of a DWC was the work presented by Wang et al. [73] using a DWC for the separation of naphthalene and cyclohexene, respectively.

In the operation and dynamics of DWCs, it is often relatively difficult to improve the composition of the side stream. In this sense, Chen et al. [74] presented a work to try to reduce this disadvantage through an over-design. Studying three case studies, they demonstrated greater flexibility in the dynamics of the schemes following the proposed methodology.

Regarding reactive DWC, Sun et al. [75] proposed a short method for the design of a reactive DWC. The methodology was based on the Underwood equations, the minimum vapor flow method, and the Vmin diagram to design a reactive DWC for the production of MTBE.

Ge et al. [76] introduced a DWC designed method based on systematic optimization using neural networks and genetic algorithms. The

method showed strengths in designing DWCs by evaluating a small portion of the possible combinations of a number of stages. The results demonstrated some improvement compared to the cases reported in the literature.

Multiple steady states have been studied by Wang et al. [77]. In their work, by using simulation analysis, four different solutions were obtained to separate the benzene-toluene-xylene mixture.

In a theoretical-experimental study, Hu et al. [78] analyzed the separation of the hexane-heptane-octane mixture in a self-made DWC. Through this study, the effects of the feeding stage, the side stream, the liquid split ratio, and the steam split ratio were explored. Optimal design values were obtained with the Kriging model and genetic algorithms. Further, their proposal could be directly applied in the experimental DWC. Another pilot plant application was presented by Zhai et al. [79], by means of theoretical analysis and the optimization of a DWC for the production of cumene was carried out. Once the optimal parameters were obtained, they were replicated in an existing DWC. The results showed savings in total annual costs.

DWC has also been used to study azeotropic mixtures. In particular, the butanol-water mixture generates a heterogeneous azeotrope. Yu et al. [80] modeled an azeotropic DWC for the separation of butanol by using cyclohexane as an entrainer. Their results revealed energy savings of 23.8% and a reduction of 19.93% in the total annual cost. Additionally, they established a control structure to reduce the effect of the disturbances.

DWCs with more than one reactive zone has been also proposed. For example, Zhang et al. [81] presented a DWC with two reactive zones as an alternative to two reactive distillation columns. As a result, they showed savings in capital cost. Another work where the DWC was considered for reactive purposes was the example presented by Dai et al. [82]. In order to produce n-propyl propynate, a DWC was designed. Savings of 12.4% were observed in energy consumption and 16.4% in total annual cost. Additionally, a control structure was established.

To improve the controllability in the compositions of the three output streams of a ternary DWC, Yuan et al. [83] introduced a feed splitting. For the case of the separation of the ethanol-propanol-butanol mixture, an improvement in the dynamics and controllability of the process was observed.

With a similar goal, Fan et al. [84] investigated a DWC to separate a quaternary mixture of n-pentane/n-hexane/n-octane/n-octane under three different control structures. The three control structures were: structure with fixed split ratios, structure with an active liquid split, and structure with an active vapor split. Their work demonstrated that traditional single-loop PID control is able to handle disturbances in the proposed DWC.

To control a reactive DWC, Qian et al. [85] put forth four control schemes based on composition and temperature controllers, under disturbances of 20% in the feed flow and the feed composition. Subsequently, Qian et al. [86] used a model predictive control to control a reactive DWC. The comparison of their strategy was carried out with a PI controller, reducing deviation, oscillations, and settling times. On the other hand, Wang et al. [87] analyzed the design and controllability of an extractive dividing wall column and pressure-swing distillation for separating an azeotropic mixture of acetonitrile/n-propanol. The design strategy was based on minimizing the total annual cost. The results showed that the pressure-swing distillation has the advantages of economic savings and control stability compared with the extractive DWCs.

To improve the energy performance of a DWC, Feng et al. [88] proposed an energy-efficient distillation technology by combining a reactive DWC with vapor recompression. The results demonstrated that the reactive DWC with vapor recompression and an intermediate reboiler is more energetically attractive than considering vapor recompression and a conventional reboiler. Xu et al. [89] also implemented vapor recompression techniques. However, their proposal tried to provide a numerical methodology to choose in a simple way, the location of the position of the heat exchanger for vapor recompression. The column

grand composite curve profiles were used to determine the type of phase withdrawn from the side product stage.

In a study which addressed a similar problem, Xu et al. [90] presented novel schemes with vapor recompression to fully recover the heat generated by the VRC under a large compression ratio. In their intensified configurations, the reboiler is used to vaporize the side liquid stream in an intermediate reboiler, and the overhead vapor is preheated by the subcooled liquid. The best intermediate reboiler location was found using the aid of the column grand composite curves of the DWC.

In an interesting combination of a reactive DWC with vapor recompression, Shi et al. [91] presented this alternative for the production of methyl-acetate and 1,3-dichlorohydrin. Through their studies, it was found that implementing both intensification techniques can substantially reduce the consumption of utilities. Also, Yang et al. [92] introduced a similar proposal for the production of TAME. The results obtained by simulation showed a saving of 43.58% in the total annual cost in the production of TAME with a mole purity of 99.958%.

As an experimental alternative, Li et al. [93] carried out research on an azeotropic reactive DWC for the production of ethyl acetate. Initially, the column was modeled and simulated, later, the simulation results were used to operate the experimental column. The theoretical results were in agreement with the experimental results. An improvement in energy requirements was observed compared to the traditional reactive distillation and the azeotropic-reactive DWC.

Yuan et al. [94] introduced a feed thermal condition adjustment strategy, which was achieved by the installation of a pre-heater in feed pipeline, in order to eliminate the black-hole problem and to enhance process flexibility and operability. Through simulations in steady-state and dynamic fashions, it was concluded that the thermal adjustment in the feeding conditions increased the flexibility and operability of the DWC.

To treat a five-component mixture, Zhang et al. [95] conducted research where a DWC was analyzed for a mixture of five components. The model for that column was created using dynamic programming to minimize the total annual cost. Based on the optimal distillation sequence obtained, the impact of intermolecular forces was found to be instructive to reduce optimization computation effort.

Ge et al. [96] investigated the thermodynamic equivalence of a DWC with two walls compared to a conventional distillation sequence for multicomponent mixtures. Additionally, in order to improve the controllability of their proposal, the vapor-liquid thermal coupling streams between column sections were transferred into a liquid-only stream. It was also proven that the derived configuration was thermodynamic equivalent to the original ones in minimum vapor flow conditions.

The ease of separation index (ESI) is defined as the ratio of the relative volatility between the light and heavy components. Wang et al. [97] presented a proposal for new generalized ease of separation index (GESI) to include the separation requirement into consideration for the selection of the optimum ternary distillation configuration. They confirmed through numerical simulation that, for a given ESI, a part of the partition at the triangle map took place with the extrade in GESI. Additionally, for a given GESI, moderate adjustments of the partition at the ternary map will be discovered with the ESI.

Due to the presence of multiple steady states, Song et al. [98] presented a DWC design alternative considering the multiple steady states. Once the DWC is optimized, extra steps are added to verify the existence of multiple steady states to later select the best design.

Feng et al. [99] studied a reactive DWC for the production of methyl acetate and isopropanol. They used three strategies to control internal liquid flows through the wall. The results showed that the control structure where the flowrate of the internal liquid stream is ratioed to the liquid flowrate of the tray of the column exhibited the best performance. In another controllability study, Wang et al. [100] applied control strategies using model predictive control and PI controllers in a DWC to separate ethanol-propanol-butanol. The results demonstrated

that both strategies can control the disturbances, however, the model predictive control strategy was more stable and with better performance.

Through a graphic analysis of the residual curves, Yang et al. [101], investigated the feasibility and restrictions of a heat integration-extractive dividing wall column for the separation of a heterogeneous methanol-toluene-water mixture. Using global optimization and dynamic analysis in a simulator, a reduction of 15.14% in energy requirements was achieved compared to the optimal double-column extractive distillation with an additional decanter. In the same sense, Wang et al. [102] proposed a thermally coupled extractive distillation sequence based on extractive DWC for separating a multi-azeotropic mixture of acetonitrile-benzene-methanol. Their results indicated reductions of 38.44%, 32.84% and 38.41% in terms of total reboiler duty, reduction of TAC and CO₂ emissions respectively.

In recent years, a considerable number of reactive DWC investigations have been conducted. For example, Li et al. [103] proposed the use of a DWC for the co-production of ethyl acetate and n-butyl acetate. In this process, the n-butyl-acetate was a product but also an entrainer to remove the water generated in the esterification reaction. In an experimental column, the data obtained in a commercial simulator were verified, having economic savings of 20.4% in the total annual cost compared to the traditional process. In the same manner, Yang et al. [104] proposed a reactive DWC combined with vapor recompression for the production of diethyl carbonate. Their work showed a reduction in the total annual cost of 20.5% compared to the conventional process.

In a combined design and control study, Pan et al. [105] presented a reactive DWC for the production of n-butyl acetate. A controllability study was performed using PI controllers. Additionally, the safety of the DWC was analyzed and the results indicated that the best control structure is the one that in the safety analysis was the worst alternative.

Li et al. [106] carried out a study on three self-heat recover reactive DWCs. The study considered a pinch-type analysis for proper energy use. Using their technique, it was possible to reduce 58% of energy consumption compared to the conventional alternative.

In another work, Qian et al. [107] studied a feasible control structure to control a quaternary Kaibel DWC. Two temperature control structures, two temperature difference control structures, and two double temperature difference control structures were studied. Their results illustrated that temperature difference control was better than temperature control to maintain product purities. Another control study presented by Qian et al. [108] demonstrated the effect of using temperature difference control and pressure-compensated temperature difference control for four-product extended dividing wall columns. Using the methanol-ethanol-propanol-butanol mixture as a case study, the results showed that the temperature difference control showed better abilities to handle disturbances.

Considering the multiple steady states of the DWC and their effect on operating costs, Song et al. [109] presented a study where the effect of the reflux ratio on multi-steady states in the thermally coupled distillation column was investigated. Additionally, based on the dynamic simulation, a new approach to select the real best design and operational parameters for the chemical process was found.

Recently, Yuan et al. [110] proposed an effective derivation of asymmetrical temperature control (ATC) schemes for DWCs. In terms of the operation of a benzene-toluene-o-xylene DWDC, the proposed method is assessed by means of a thorough comparison between the derived ATC scheme and the double temperature difference control scheme. In comparison with the latter, the former displays not only relatively better transient responses but also smaller steady-state deviations in the three controlled product qualities. These findings demonstrate the effectiveness and feasibility of the method proposed for deriving ATC schemes.

Li et al. [111] studied how to use a hybrid reactive DWC for the production of n-butyl acetate. This proposal considered the usage of a polyoctylmethylsiloxane pervaporation membrane to replace a

methanol recovery column. By implementing this hybrid process, savings in operating costs of 40.8% and 32.6% were achieved in the total annual cost.

Considering a multiple substitutions of conventional couplings for liquid splits, Cui et al. [112] investigated the dynamic performance of a double liquid-only side-stream process. To improve the effectiveness of the inferential control scheme, a modified structure with one internal composition controller was proposed. The result of this work encouraged the promotion of liquid-only side-stream configurations, as a competitive substitute of the well-known Petlyuk column.

In another control study, Ge et al. [113] put forth an easy-to-operate and energy-efficient four-product dividing wall columns with two partition walls. The consideration was that the bidirectional vapor-liquid thermal coupling streams between column sections were converted to liquid-only transfer streams to obtain new variants. As a result, mitigation of the energy requirements and improvement in the little controllability of the steam splits was observed.

2.4. Latin America

In Latin American regions other than Mexico, relatively few papers have been published regarding the DWC. A study which addresses reactive DWC was reported by Santaella et al. [114]. They compared different technologies for the production of ethyl acetate. These technologies considered for the production of this compound were conventional process, reactive distillation, reactive distillation with pressure swing, dividing wall column with reactive reboiler. Evaluating with sustainability indicators, reactive DWC turned out to be the best alternative showing savings of 46% in energy and 26% cost savings compared with the traditional process.

From the same research group, Santaella et al. [115] presented as an alternative a reactive DWC for the production of triethyl citrate. The intensified process was compared to the traditional reaction-separation route. As a result, final designs showed citric acid conversions above 99.9%, energy consumption from 3-5 MJ / kg, and a TEC production costs of 1.5 USD / kg.

Cordeiro et al. [116] introduced a systematic strategy of a DWC applied to an extractive distillation process in their research. The best results in terms of the total annual cost (TAC) were obtained for columns with a distinct number of stages in each section of the wall; however, these columns did not outperform the optimized conventional systems.

On the other hand, Junqueira et al. [117] presented six alternatives for the production of cumene: the conventional process, transalkylation, heat-integrated, dividing wall column, reactive distillation and double-effect distillation technologies. They used two performances indexes in order to evaluate all six alternatives. The economic analysis was performed by estimating their respective gross annual profit, whereas the environmental assessment was carried out by calculating seven eco-indicators. The study showed that the intensified processes are not only more economically attractive but also more environmentally friendly.

Finally, Biasi et al. [118] proposed a unified model to simulate columns with division of multiple phases, including the DWC. Their proposal model uses a unique set of MESH-equations for parastillation, metastillation, and conventional distillation. All columns reduce the operational and total annual costs in 19% and 15%, when compared to conventional columns.

2.5. DWC in México from Professor Arturo Jimenez Group or Professor Arturo's Formed Students

Regarding DWC, when the papers are associated to institutions in the world, the Universidad de Guanajuato has published approximately 55 and the Instituto Tecnológico de México in Celaya has published 28. It is important to highlight that the research group in the University of Guanajuato was formed by Professor Jiménez from the Instituto

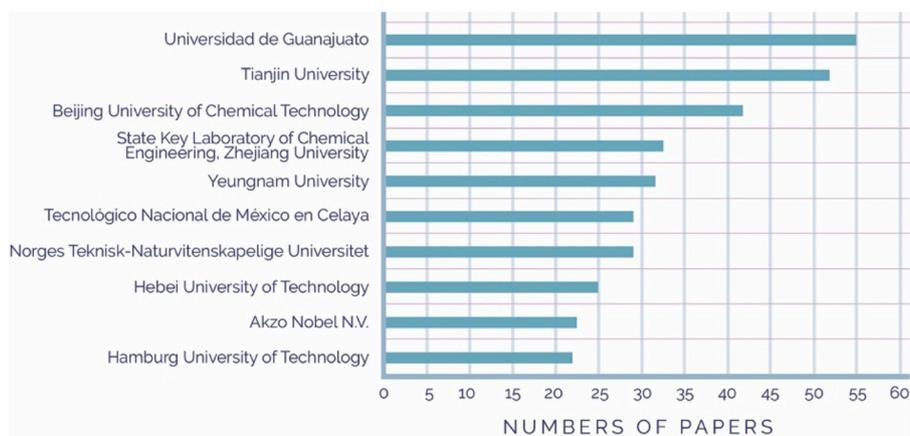


Fig. 4. Research papers published by institutions in the world, regarding the topics of Petlyuk column or dividing wall column according to a search conducted in the Scopus-Elsevier data base (September 16, 2020).

Tecnológico de México in Celaya. Fig. 4 shows the research papers published in different institutions throughout the world in relation to the topic of Petlyuk column or dividing wall column.

In this paper we would like to highlight, in a short review, the research carried out particularly in Mexico by Prof. Jiménez and his research group (Instituto Tecnológico de México in Celaya) who have carried out the pioneering work of DWC. Throughout the years, the PhD students who have been advised by Prof Jimenez with his respective research groups have continued and expanded the lines of work on the topic. It should be noticed that in Latin America, Mexico is the only country where substantial development has been generated regarding the DWC and its various approaches (Figs. 3 and 4). In the following paragraphs, a series of works that were developed from the ideas from Professor Jimenez will be briefly presented. This valuable research has as a central axis direct applications of the DWC, as well as complementary studies that serve to have a broad overview of the capabilities of the DWC

One groundbreaking work in Mexico regarding the DWC is the paper by Hernandez and Jimenez [119]. In this work, a strategy for the energy-efficient design of the fully thermally coupled distillation column (Petlyuk system) is presented. The strategy is based on a dynamic model and uses two recycle streams as design variables in order to detect the minimum energy supplied to the reboiler. The importance of the reported methodology is that the dynamic model can be used for design and control studies.

In 2001, Jiménez et al., [120] presented a controllability analysis of seven distillation sequences for the separation of ternary mixtures using the singular value decomposition technique and closed-loop responses under feedback control. The results showed that nonconventional distillation sequences, such as the Petlyuk column, can have better control properties than nonintegrated schemes. This result is important because it was expected control problems due to the presence of recycling streams.

A similar study regarding ternary mixtures was conducted by means of the controllability properties of thermally coupled distillation sequences (including the Petlyuk column) which were compared with those of the conventional direct and indirect sequences by Segovia-Hernandez et al. [121]. Closed-loop responses to setpoint changes were performed, and controllers were tuned to minimize their ISE values. The results indicate that the integrated systems exhibit better control properties than sequences based on conventional distillation columns.

Blancarte-Palacios et al. [122] extended the design procedure for thermally coupled distillation sequences for the separation of four-component mixtures. The schemes analyzed include a sequence with a prefractionator (Petlyuk-type column). The results corroborate

energy savings of approximately 30% for the complex distillation sequences in the separation of some quaternary mixtures of hydrocarbons.

Continuing with studies that assess the control properties of dividing wall columns under the light of conventional separation schemes, Segovia-Hernández et al. [123] analyzed the control properties of six alternatives thermally coupled distillation schemes to the Petlyuk system. The theoretical control properties are analyzed with the application of the singular value decomposition technique. Rigorous closed-loop simulations are used to supplement the theoretical analysis. The results indicate that a reduction in the number of interconnections of the Petlyuk configuration does not necessarily provide an improvement of its controllability properties.

One of the first works of the study of DWC with non-equilibrium models was reported by Abad-Zarate et al. [124]. The results generated by the non-ideal model are in agreement with data reported in the literature. Hernández et al. [125] and Tamayo-Galván et al. [126] studied the thermodynamic equivalence and the control properties of six equivalent schemes to the DWC. Despite the simplicity of thermodynamically equivalent schemes, the DWC shows operational and controllability advantages.

In 2007, a PI controller with a dynamic estimation of uncertainties is implemented for the control of the Petlyuk column by Segovia-Hernández et al. [127]. Comparison with the classical PI control law was carried out to analyze the performance of the proposed controller in facing unknown load disturbances in feed composition and setpoint changes. The results show that the implementation of the controller with a dynamic estimation of uncertainties improved noticeably the closed-loop responses provided by the PI controller.

The design of the DWC with trays is important for systems with high vapor loads. Thus, a strategy for the mechanical design of sieve trays in a dividing wall column is presented in the work by Rodríguez-Angeles et al. [128]. Furthermore, an operational analysis of the trays using computational fluid dynamics (CFD) is reported. Designed trays are tested in terms of weir flooding, active zone flooding, and flow regime. A reported strategy allows obtaining operational designs for the trays of the whole column.

A systematic technique to tune PI controllers for a class of DWCs with periodic discrete measurements was developed by Zavala-Guzmán et al. [129]. Providing classical parameters of the process dynamics (static gains and time constant of open-loop response) and the sampling-delay time of measurements, this technique determines effective gains for every controller of the DWC, in a straight forward and simultaneous way.

The separation of a multicomponent hydrocarbon mixture in a sequence involving one or more DWC was proposed by Lucero-Robles et al. [130]. Sequences were analyzed in a simulation environment

and compared in terms of total energy requirements, environmental impact, and controllability. The location of the DWC in the separation train depends on the properties of the mixture to be purified, and affects the control properties and energy consumption.

Tututi-Avila et al. [131] focused on the design, dynamics, and control of an extended dividing wall distillation column, referred to as a satellite column, and its comparison to the performance of a DWC column and to a direct distillation sequence for the separation of BTX mixtures. The results from the optimized designs for these distillation structures show that the satellite column is the most energy-efficient configuration, as far as the dynamics are concerned. The satellite column shows a similar performance when compared to the DWC column and to the conventional direct separation sequence, indicating that the additional energy savings provided by the satellite configuration can be achieved without deterioration of its control behavior.

The dynamic performance of a dividing-wall-based structure for the separation of a five-component mixture is studied by Segovia-Hernández et al. [132]. A sensitivity analysis is performed on the structure in terms of the interlinking streams, performing a singular value decomposition analysis to selected cases with different operational conditions. The designs with the lowest energy duties also showed the best open-loop properties.

Castillo-Landero et al. [133,134] presented an intensification methodology that minimizes the number of equipment units required for the transformation of raw materials into products. It is shown how an original flowsheet with one chemical reactor and three distillation columns is gradually transformed into an intensified process (DWC) that provides an alternative with superior economic and sustainability metrics.

The quaternary dividing wall column has not been considered as an option, and currently is not a well explored option since its architecture and dynamic properties have not been completely studied. Sánchez-Ramírez et al. [135] carried out research on a set of quaternary dividing wall columns. They were designed and tested in many performance indexes: energy requirement, environmental impact, inherent safety, and dynamic properties. The interesting approach of this proposal is that many of the conventional thermal couplings are substituted for liquid splits whose implementation improves the performance indexes already mentioned.

The optimal design of dividing wall columns is a non-linear and multivariable problem, and the objective function used as optimization criterion is generally non-convex with several local optimums. Considering this fact, several relevant works have been published on this topic, for example, Gomez-Castro et al. [136], Gutiérrez-Antonio and Briones-Ramírez [137], and Vazquez Castillo et al. [138] studied the design of dividing wall columns, for separation of ternary and quaternary mixtures, using as a design tool, a multi-objective genetic algorithm with restrictions, written in Matlab and using the process simulator Aspen Plus for the evaluation of the objective function.

An optimization approach for DWC for the separation of ternary mixtures is presented by Ramírez-Corona et al. [139]. The approach uses a shortcut design method that allows the system to be modeled as a nonlinear programming problem. Several cases of study show the applicability of the proposed approach.

Vázquez-Castillo et al. [140] and Cabrera-Ruiz et al. [141] introduce a multi-objective optimization approach to integrate the design and control of multicomponent distillation sequences. It has been found in this approach that the dividing wall distillation column exhibits better control properties than the conventional separation systems. In addition, the environmental aspects of the designs, in particular their environmental impacts together with their control properties, can be established.

López-Saucedo et al. [142] address the optimization of a nonconventional dividing wall batch distillation column with and without chemical reaction. The simultaneous solution of these systems of differential and algebraic equations is performed using two different

approaches: pure equation oriented approach based on orthogonal collocation over finite elements and control vector parameterization.

Over the years the lines of research have been expanded to study complex applications at the DWC. For example, one of the first works in this line is the paper by Bravo-Bravo et al. [143]. The authors present a novel extractive dividing wall distillation column, which has been designed using a constrained stochastic multiobjective optimization technique. The approach is based on the use of genetic algorithms to determine the design that minimizes energy consumption and total annualized cost. Several case studies are used to show the feasibility of performing extractive separations in DWCs. In a similar context, Miranda-Galindo et al. [144] have studied the design of reactive Petlyuk distillation column (using as a study case the production of fatty esters), generalizing the use of a multiobjective genetic algorithm with restrictions coupled to Aspen Plus.

Bravo-Bravo et al. [145] researched a hybrid distillation/melt crystallization process, using conventional and the DWC. The design and optimization were carried out using, as a design tool, a multi-objective genetic algorithm with restrictions coupled with the process simulator Aspen Plus, for the evaluation of the objective function. The results show that this hybrid configuration with DWC is a feasible option in terms of energy savings, capital investment, and control properties.

The DWC based on cryogenic extractive distillation was proposed and simulated by using Aspen Plus coupled to a multi-objective stochastic optimization procedure (differential evolution) was analyzed by Torres-Ortega et al. [146]. The evaluation of the performances of the proposed configurations focused on the ethane-carbon dioxide azeotrope separation considering different liquefied hydrocarbon fractions as entrainers. The design alternatives were compared to the conventional chemical absorption system. The proposed cryogenic extractive DWC sequences realized the carbon dioxide removal together with the lower TAC compared to the conventional chemical absorption system.

As a result of various basic engineering studies regarding the DWC, the design, construction, and operation of a prototype of a dividing-wall distillation column were reported by Hernández et al. [147], Barroso-Muñoz et al. [148], Barroso-Muñoz et al. [149] and Delgado-Delgado et al. [150]. These studies report the implementation of an experimental DWC, being the first one reported in Latin America. The reaction between ethanol and acetic acid was conducted within the prototype, and the experimental results indicate that a heterogeneous mixture of ethyl acetate and water is obtained as the top product. The temperature profile measured during the experimental run can be used for controlling the batch distillation column in cyclic operation mode. In the same prototype the production of Methyl Oleate was studied by López-Ramírez et al. [151].

In recent years, the use of DWC in the production and purification of biofuels and bioblocks has been examined with significant energy savings and reduction in total annual costs, in comparison with conventional distillation configurations: biodiesel [152,153], bioethanol [154–156], biobutanol [157,158], jet fuel [159,160], furfural [161], levulinic acid [162], ethyl levulinate [163], 2,3-Butanediol [164], lactic acid [165], among others.

The pioneering lines of research in the DWC study developed by Prof. Jimenez and his research group (and over the years continued and expanded by various groups of researchers in Mexico) have allowed the study of DWC and complex distillation columns to be atopic of high relevance in our continent. Some representative works recently developed in Latin America represent this relevance.

Santaella et al. [115] describe a systematic process design for triethyl citrate production by direct esterification of citric acid with ethanol via simultaneous reaction-separation technologies. The design methodology includes the simultaneous optimization of controllability and profitability criteria. The reactive dividing wall column scheme results in large energy and cost savings over a traditional reactive distillation process.

Rawlings et al. [166] carried out a study regarding the modeling,

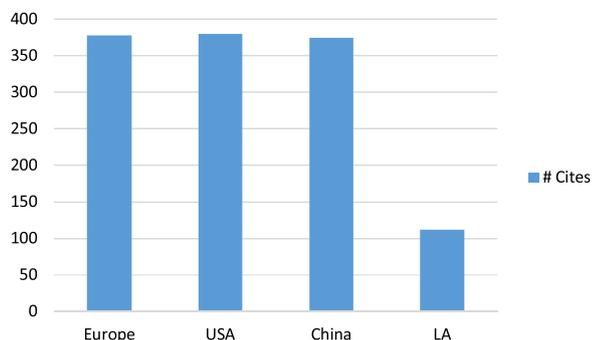


Fig. 5. Cited documents from Professor Arturo Jimenez in the world according to a search conducted in the Scopus-Elsevier data base (September 16, 2020).

optimization, and conceptual design of a dividing wall column for the separation of four products by implementing three different formulations: an NLP, an MINLP, and a GDP formulation. For its solution, the authors propose a rigorous tray-by-tray model and compared the results with commercial software, followed by its reformulation to include a mixed-integer nonlinear programming and a general disjunctive programming formulation to respond to the conceptual design problem attached to these complex configurations.

A methodology for gradual process intensification (using DWC) is applied to a conventional flowsheet for the production of the green solvent ethyl lactate from ethyl alcohol and lactic acid by Tusso-Pinzon et al. [167]. The approach takes the base design and integrates two pieces of equipment into one at each step of the intensification task. Each intensified structure is rated through economic, environmental, sustainability, and inherent safety metrics.

It should be noted that there is a great deal of work related to the DWC. Regarding this, the research in which Professor Jimenez has participated has had a considerable impact throughout the world. A significant sample is shown in Fig. 5 where the number of citations of Professor Jimenez by geographic regions is observed, without considering the citations generated in Mexico.

In summary, the documents generated from the work of Professor Jimenez have to date a total of 3,059 citations, thus having an *h*-index of 33, as reported in Scopus until September 16, 2020. Additionally, Professor Jimenez has trained approximately 20 Ph.D. students and 58 Master's students, many of them also human resources producers.

3. Conclusions

The distillation columns have proved to be the most reliable separation method. However, as the number of components to be separated increases, the number of columns and the energy required to run these columns also increase. This results in huge capital costs that are unaffordable due to limited energy resources. A most "compact" column appeared to solve this difficulty and this column is called the divided wall column which is capable of separating mixtures of three or more components to high purity products with energy less than the conventional process. The DWC is the most widely practiced distillation technique among the various energy-efficient distillation systems available, and many studies have examined its energy-saving performance, design, optimization, and control behavior. In Latin America, particularly in Mexico, the research group of Prof. Jiménez is a pioneer in the comprehensive study of this complex and intensified distillation system. Even considering the region of Latin America, the first experimental DWC reactive originated in Mexico. Moreover, the contribution and impact that their contributions have generated are reflected in the number of citations of their works, with an approximate total of 1,243 citations in regions outside of Mexico. His academic contributions can be summarized in 164 documents, 3,059 cites with an *h*-index of 33 according to scopus. Over the years, different research groups in Mexico

(all of them formed directly or indirectly by Prof. Arturo Jiménez) have expanded the studies and these studies provide substantial contributions to the state of the art of the DWC, which can be appreciated from the large number of published papers published and the citations.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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