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Introduction

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1.1 Process Intensification

In recent years, process intensification has been one of the most active areas of research in chemical engineering. It refers to the use of novel equipment and/or methods to drastically improve the performance of a process or unit. Process intensification could result in a substantial reduction in equipment size, a reduction in energy consumption, an increase in product yield, a reduction in waste, or anything that ultimately leads to economical and sustainable technologies (Stankiewicz and Moulijn, 2000). It is dissimilar to merely scaling up the plant to make it more economical. One of the most important features of process intensification is that the changes it brings are drastic in nature and are revolutionary rather than evolutionary (Stankiewicz and Drinkenburg, 2004).

The advent of process intensification in the chemical engineering field was marked in 1983, when the very first paper was published on the application of centrifugal fields (i.e. "HiGee," also known as rotating packed bed) in distillation processes (Ramshaw, 1983). In the 1980s, process intensification was mainly referred to as a substantial reduction in the size of the equipment (Stankiewicz and Drinkenburg, 2004). The first definition of process intensification by Ramshaw is "devising an exceedingly compact plant which reduces both the 'main plant item' and the installations costs" (Ramshaw, 1983).

The definition of process intensification has changed, and now it is not limited only to the substantial reduction in size. Currently, process intensification refers to novel equipment and techniques that lead to inexpensive and sustainable processes. At present, conventional methods are becoming unsustainable because of the stricter environmental policies and agreements across the world. One of the definitions of process intensification that is deemed suitable in the current era is "Any chemical engineering development that leads to substantially smaller, cleaner, safer

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and more energy efficient technology or that combine[s] multiple operations into fewer devices (or a single apparatus)" (Baldea, 2015).

Gerven and Stankiewicz (2009) reported four main goals of process intensification:

1. Maximize the effectiveness of intermolecular and intramolecular events.
2. Deliver the same physical and chemical environment to the molecules.
3. Optimize the driving forces of the process and increase the specific surface area.
4. Maximize the synergy of partial processes.

The four approaches to achieve the above goals are reported as follows (Gerven and Stankiewicz, 2009):

1. The first approach is structure (spatial domain); it provides different explicit spatial structuring into different equipment or reactant assemblies, which can achieve the above goals. For example, static mixers with a specific structure, heterogeneous catalysts with a specific structure/shape, etc. to achieve goals 1, 2, and 3.
2. The second approach is energy (thermodynamic domain), which provides efficient energy distribution and utilization of different forms of energy available to attain the above goals. For example, ultrasound/microwave-assisted reactors where all molecules experience the same physical and/or chemical environment to achieve goals 1, 2, and 3).
3. The third approach is synergy (i.e. functional domain), in which various phenomena function synergically to achieve goals 1, 3, and 4. An example of this is reactive distillation (RD).
4. The fourth approach is time (i.e. temporal domain), which provides control over time scales. For example, an oscillatory baffle flow reactor facilitates controlled energy input.

Stankiewicz and Drinkenburg (2004) presented a toolbox to achieve process intensification. It includes equipment and methods for process intensification. The former dealt with the change in hardware to achieve intensification, whereas the latter dealt with the change in the way a process is carried out (software) to achieve intensification.

Currently, climate change is one of the major concerns, and chemical/process industries are considerably responsible for it. These industries continue to contribute to the disruption of the environment, biosphere, biochemical flow, and land systems (Steffen et al., 2015). These have prompted the formation of several regulations, international/national treaties, procedures, and consumer preferences that seek sustainable alternatives. The reduction of greenhouse gasses' emissions, addressal of resource paucity, and revisiting material management are among many efforts that may enhance sustainability. These sustainable changes can be achieved through integrated innovation, which is interrelated with process intensification (López-Guajardo et al., 2022).

Process intensification is well recognized in current trends because of its synergy with Industry 4.0 and circular chemistry (López-Guajardo et al., 2022). The objective of circular chemistry is to (re)investigate the better use of resources by decreasing the consumption of the resources, improving efficiency, increasing the life of products, and maintaining them in the production cycle (Keijer et al., 2019). Industry 4.0, conceptualized in the year 2011, revolutionizes the way different process systems function within an integrated framework. This is related to the application of new and advanced technological structures, for example, the Internet of Things (IoT), big data and analytics, artificial intelligence (AI), cloud computing, and cybersecurity (Sharma et al., 2021). The technological advancements have modernized conventional industrial operations, procedures, and instruments (Canas et al., 2021), leading to improvement in the overall process of circularity (circular economy) by means of adopting sustainable practices. Thus, a clear overlap is present between the benefits of Industry 4.0 and the main objectives of process intensification. To achieve sustainable development, López-Guajardo et al. (2022) suggested a process intensification 4.0 strategy, which is the confluence of Industry 4.0, circular chemistry, and process intensification.

1.2 Need for Control and Safety Analysis of Intensified Chemical Processes

Despite the numerous benefits of process intensification, the controllability and safety of the intensified processes must be evaluated, as intensification may introduce certain specific hazards into the process. In the drive towards newer and/or better processes, industries should ensure that new hazards are not created on account of process intensification. Potential problems due to process intensification include the following (Etchells, 2005):

- Some process intensification technologies require excessive electricity inputs (e.g. for microwaves, excessive voltages, or electromagnetic radiation) or must be operated at higher temperatures and/or pressures. The high-energy sources may introduce new hazards that have to be considered when applied to hazardous substances, e.g. whether it is safe or not to use microwaves on thermally unstable substances or mixtures.
- Control and safety of intensified systems can be challenging. Although process intensification may reduce hazards (e.g. by substantially reducing the material inventory), control degrees of freedom may be reduced due to intensification. This reduction may affect the controllability of the process.
- Due to better mixing, intensified reactors have the potential to dramatically increase reaction rates. In comparison to conventional reactors, this might result in a substantially higher rate of energy release in some cases, and it might also, in some situations, affect the reaction chemistry. If the reaction has not been

properly evaluated before, it could have serious safety implications (e.g. if the increased reaction produces a gas rather than a liquid). While there are tested and proven methods for determining the anticipated reaction thermochemistry in conventional reactors, these methods are less reliable for some recent types of intensified equipment (Etchells, 2005).

- Rotating equipment, which may be introduced on account of process intensification, may not be appropriate for materials that are friction sensitive, or those that could ignite or explode under friction.
- Hybrid or integrated intensified systems will invariably have many smaller parts with more connections. This will increase the chances of seal leakages due to the greater number of joints.
- Heat integration with mechanical vapor recompression may result in potential fire and explosion hazards if the gas compressor seal fails.
- The short residence time in intensified processes requires control instruments that respond rapidly and detect deviations (Klais et al., 2009).

The abovementioned factors require a careful analysis of the controllability and safety of the intensified process, without which the intensified process, though efficient, may become infeasible to operate. At various stages of design and operation, the risk of accidents has to be investigated. Chemical process safety can be broadly classified into four tiers of protection, as presented in Figure 1.1 (Ebrahimi

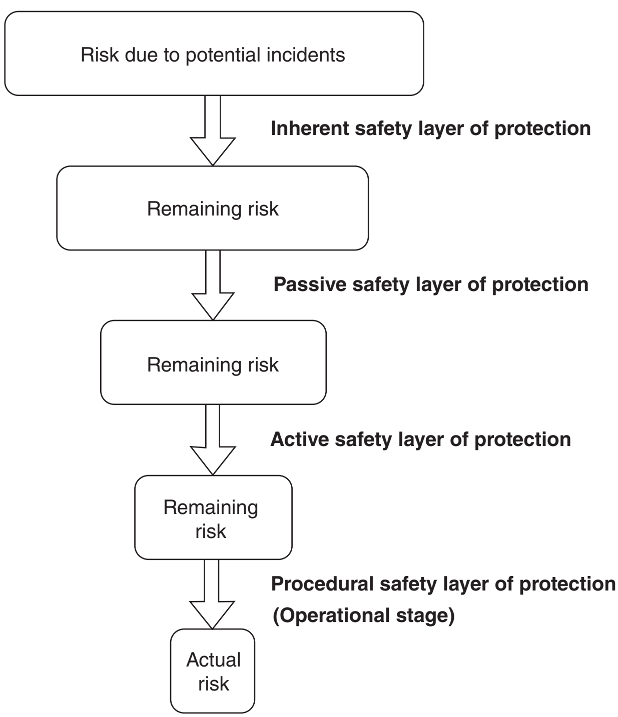

Figure 1.1 Layers of protection for a chemical process and their effect on reducing the risk.

et al., 2012). Even if the inherent safety layer is frequently the most effective one, it is only logical to have additional layers of protection. It is rarely probable that all these levels will fail simultaneously.

1.3 Studies on Control and Safety Analysis of Intensified Chemical Processes

In this section, we analyze the studies on the control and safety of intensified chemical processes in the last two decades. First, the Scopus database was searched on 7 September 2023, for articles containing "intensification or intensified" in their title, abstract, and/or keywords; this search resulted in a total of 142,492 articles in all subject areas, without any limitations. Out of these entries, 8470 articles (journal papers, books, and book chapters, while excluding conference papers, review articles, notes, editorials, etc.) in all languages are in the chemical engineering field from the year 2000 to 2022. These include some articles unrelated to process intensification; on the other hand, they do not include some articles related to process integration, such as those on improving the energy efficiency of distillation systems. Overall, 8470 is a reasonable estimate of articles related to process intensification. Figure 1.2, based on these articles, clearly indicates that there has been an increasing interest and research in process intensification in chemical engineering. As evident from the sharp rise in the number of published articles from the year 2016 to 2022, process intensification has received significant interest from researchers in recent years.

Subsequently, to understand the research interest and contributions on control and/or safety of intensified chemical processes, the Scopus database was searched for articles containing "intensification OR intensified" AND "control OR safety" in their title, abstract, and/or keywords; the rest of the criteria are as mentioned in the above paragraph. This resulted in 1148 articles, which are assumed to cover the control and/or safety of intensified chemical processes. The search results (Figure 1.3)

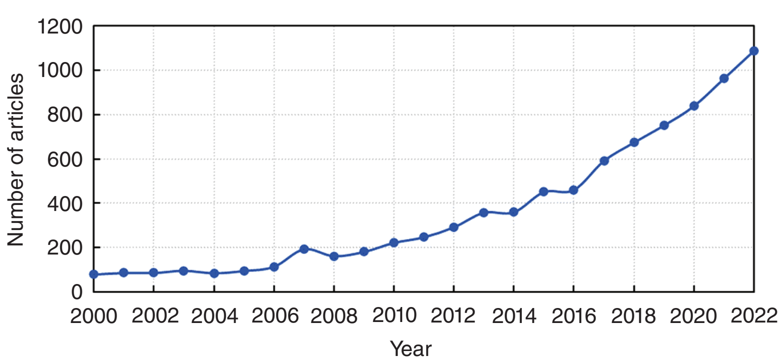

Figure 1.2 Number of articles (journal research articles, books, and book chapters) with *intensification OR intensified* in the article title, abstract, and/or keywords, published in all languages in the chemical engineering field, in each year from the year 2000 to 2022.

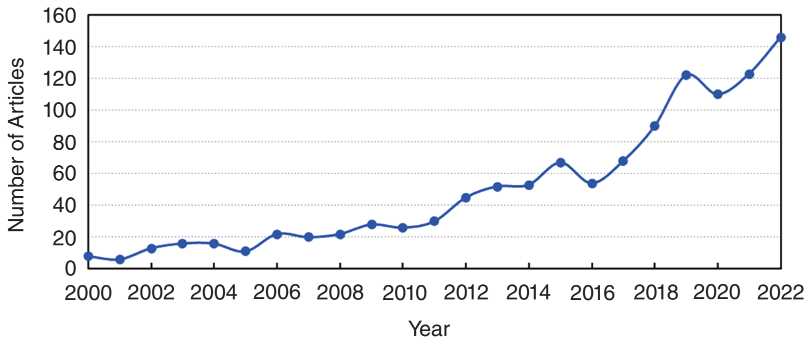

Figure 1.3 Number of articles (journal papers, books, and book chapters) annually published from the year 2000 to 2022 on control and/or safety of intensified chemical processes.

confirm that there has been a gradual and steady increase in the research on the control and/or safety of intensified chemical processes since the year 2000; particularly, the increase has been significant in recent years except for a dip in the year 2020.

The 1148 articles on control and/or safety of intensified chemical processes were published in more than 150 journals or book chapters from the year 2000 to 2022. Of these articles, 92.9% are research articles, 6.1% are book chapters, and 1% are books. The top 10 journals that published a higher number of these articles are presented in Figure 1.4. Among them, the maximum number of articles has appeared in the Chemical Engineering and Processing: Process Intensification journal, which is

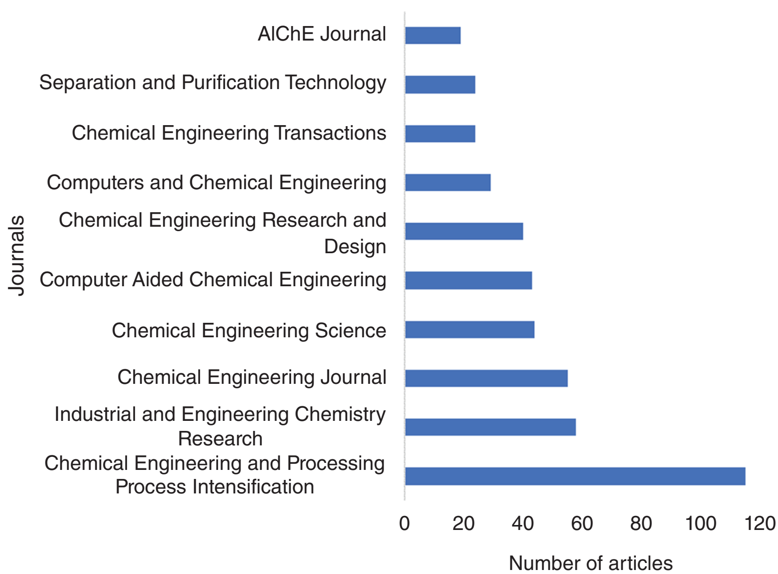

Figure 1.4 The journals that published the highest number of articles on control and/or safety of intensified chemical processes from the year 2000 to 2022.

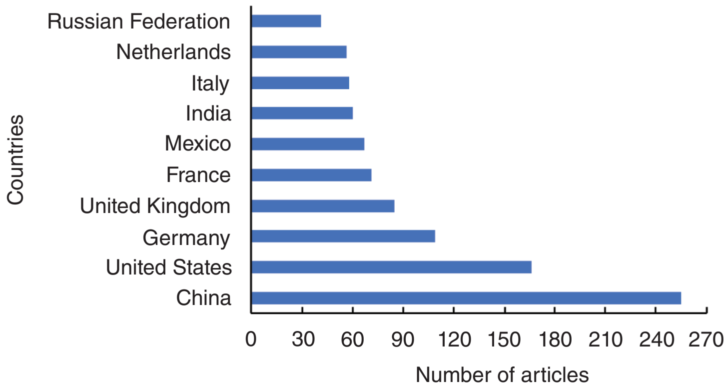

Figure 1.5 The countries that contributed the highest number of articles on control and/or safety of intensified chemical processes from the year 2000 to 2022.

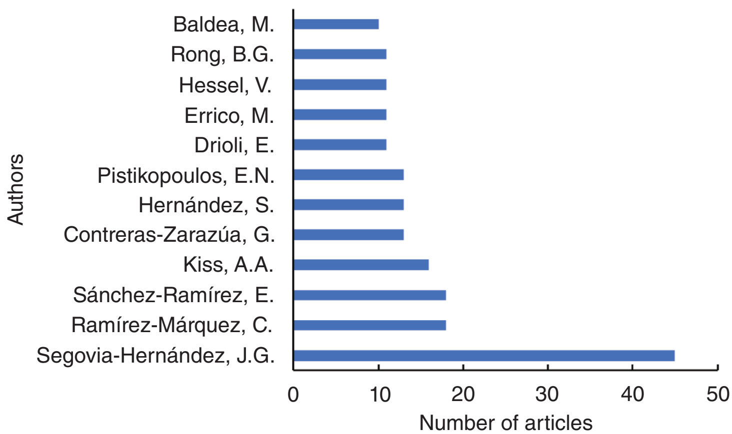

Figure 1.6 Researchers, who published 10 or more articles on control and/or safety of intensified chemical processes from the year 2000 to 2022.

not surprising. As shown in Figure 1.5, the largest number of articles on control/safety analysis of intensified processes have come from China, followed by the United States, Germany, the United Kingdom, and others. Finally, Figure 1.6 lists the 12 active researchers, each of whom has contributed 10 or more articles; in particular, Prof. J.G. Segovia-Hernández is the most active researcher with 45 articles. The editors are pleased that five of these active researchers have contributed chapters to this book.

1.4 Scope and Organization of the Book

For better understanding and for the convenience of novice readers, researchers, as well as practitioners, this book is divided into three parts. Part I (i.e. this chapter and Chapter 2) provides an overview of process intensification, its applications and developments, need for control and safety analysis, and industrial applications of process intensification. Chapter 2 will be of interest to all who are interested in process intensification. Chapters 3–5 in Part II focus on providing the basics and overview of steady-state simulation, optimization methods, programs and simulators, dynamic simulation and control, safety analysis, and popular methodologies and tools for safety analysis. These chapters will be of interest to young researchers, who may be new to control and safety field. Lastly, Chapters 6–12 in Part III deal with studies on control and safety analysis of many intensified chemical processes, including hybrid reactive-extractive distillation, RD, middle vessel batch distillation with vapor recompression, intensified extractive distillation column, dividing wall column (DWC), and DWC with mechanical vapor recompression. These advanced chapters in Part III will be of interest to researchers and practitioners working in the field. The scope of each chapter is outlined as follows.

Chapter 2, prepared by a practitioner with extensive industrial experience and keen interest in the latest developments, presents an overview of many process intensification techniques employed in industrial practice and discusses their relative merits and associated practical challenges. This chapter provides a brief and handy reference relating to process intensification for practicing engineers, researchers, and students.

Two experienced academicians in process simulation, optimization, and control prepared both Chapters 3 and 4. The general approach to process simulation, common commercial process simulators, and free process simulation software are outlined in Chapter 3. This is very useful for novice readers to refresh the basics and find a suitable process simulation platform for their applications. Then, the main steps in the simulation of intensified chemical processes are presented. Next, popular approaches for single- and multi-objective optimization of chemical processes and the linking of common optimizers with a commercial process simulator are described. Also, challenges in the simulation and optimization of intensified chemical processes are outlined. Finally, process simulation and optimization of intensified chemical processes are illustrated, taking DWC as an example.

Chapter 4 on dynamic simulation is important to understand and evaluate the transient behavior of chemical processes as well as the performance of the associated control system. Dynamic simulation is more complex and computationally challenging than steady-state simulation. Chapter 4 first introduces the principles and applications of dynamic simulation of chemical processes as well as the common software for dynamic simulation. Then, a detailed procedure involving five steps for dynamic simulation and control of intensified chemical processes is presented. Next, variables related to process control, typical control loop, and control degrees of freedom are outlined. Subsequently, the need for and difficulties in the dynamic simulation and control of intensified chemical processes are summarized. Finally, a case study on dynamic simulation and control of an intensified chemical process, namely, extractive DWC, is presented and discussed.

Chapter 5, prepared by three researchers with experience in chemical process safety and control, addresses the tools and procedures for detecting the positive and negative effects of process intensification on process safety. It examines the potential impacts of process intensification on process safety, considering positive effects such as reduced inventories of hazardous materials and lower energy consumption as well as negative effects such as increased process complexity and new safety concerns associated with novel technologies. Also, Chapter 5 highlights the critical role of safety considerations in the design, operation, and management of process intensification technologies in industries. Then, several case studies illustrate the application of safety analysis for implementing process intensification technologies, demonstrating how hazard identification, risk assessment, and inherently safer design principles can contribute to the safe operation of intensified processes.

Chapter 6, prepared by two active researchers in process control, outlines the progressive steps required to develop a robust control structure for the reactive-extractive distillation system for the recovery of isopropanol and diisopropyl ether from wastewater. In this chapter, several full control schemes (from the simplest temperature control to the complicated feedforward control) are presented in an evolutionary manner so that readers can grasp the advantages and drawbacks of each scheme. This chapter then proposes a suitable control scheme with triple-point temperature control that can regulate the purity of the products closer to the respective setpoint, despite several disturbances.

Three leading researchers in the design and control of distillation processes contributed to Chapter 7, which focuses on the design and control of RD columns with an application for the production of 4-hydroxybutyl acrylate. This work frames the RD columns within a process where recycle streams (to these columns) are present, as is typical in industrial practice. The control is focused on developing a plantwide strategy to achieve the material inventory (in other words, balancing the reaction stoichiometry), the desired production rate, and product purity. Several process disturbances (e.g. flowrate and composition changes) are implemented to test the proposed control structure of the plant.

Chapter 8 is prepared by four researchers with an interest in process simulation and control. It presents a study on improving the performance of middle-vessel batch distillation without vapor bypass but using a vapor recompression column for separating ternary (methanol/ethanol/1-propanol) zeotropic mixtures. It shows that single-stage vapor-recompressed middle-vessel batch distillation with a gain-scheduled proportional-integral controller is the best configuration in terms of energetic, economic, environmental, productivity, purity, and practical feasibility compared to conventional middle-vessel batch distillation.

Chapter 9 is the contribution of four researchers active in process systems engineering. It analyzes three process alternatives, namely, a conventional sequence of columns (CSCs), DWC, and DWC with multistage vapor recompression (MVR), for different safety indices, namely, individual risk, damage index, and process route index. Later, the safety prospects of the three processes are discussed based on industry experts' recommendations obtained through a survey. Similarity and dissimilarity between the safety prospects depicted by the considered indices and those suggested by the experts are discussed. Finally, a modification to the process route index is proposed to make it more comprehensive for analyzing the safety of processes and apply it to CSC, DWC, and DWC-MVR systems.

Chapter 10, contributed by two researchers active in process safety, presents a dynamic safety analysis of intensified extractive distillation processes for the separation of an azeotropic mixture of acetonitrile and water, focusing on column overpressure as the primary safety issue. For this analysis, multiple layers of protection, including basic process controls, alarms, safety instrumented systems, and pressure relief systems, are simulated to mitigate potential hazards arising from various hazardous scenarios. The effectiveness of these protection layers is then assessed using Aspen Plus for steady-state process design, Aspen Dynamics for dynamic simulation and control, and scenario-based dynamic safety analysis.

Chapter 11, prepared by five established researchers in process modeling, optimization, and control, describes the operational and safety aspects of bioprocesses, taking the separation and purification of methyl ethyl ketone, furfural, and lactic acid as an example. Various intensified separation and purification processes, such as RD and split-wall columns, are used. In this chapter, intensification techniques, proposed designs for each separation system, and the results are presented and discussed in terms of their operability and safety. The study reported in Chapter 11 shows that controllability and security are essential for the new intensified technologies and the enormous potential of developing intensified processes to make bioprocesses safer and more operable.

Finally, Chapter 12 by three researchers active in process development, safety, and control analyzes economic and safety objectives for ultrasound assisted and ionic liquid catalyzed in situ biodiesel production from wet microalgae (referred to as alternative 1), and the same process (i.e. alternative 1) is intensified by DWC and MVR (referred to as alternative 2). Both processes are modeled in Aspen Plus V10 and optimized using an MS Excel-based program for the elitist non-dominated sorting genetic algorithm. This study takes individual risk as the safety objective and break-even cost as the economic objective. One solution from the Pareto-optimal front of each alternative is chosen using the simple additive weighing method. Then, the chosen optimal solutions for the two alternatives are compared.

1.5 Conclusions

In essence, this book is intended to provide the chemical engineering academia, students, and practitioners with process intensification techniques/technologies and their influence on process controllability and safety. It is organized in such a way that readers can study one or more chapters of their interest independent of the other chapters. The contents of this book can be readily adopted as part of special/elective courses on process systems engineering, process design and integration, plantwide control of chemical processes, and safety and hazards analysis for chemical processes for under-/post-graduate students. Manufacturers are likely to continue to intensify the existing processes even further owing to the rising competition and regulations. This book will provide them with a comprehensive understanding of the effects of process intensification on control and safety, both of which are indispensable. Finally, it is hoped that this book will be valuable to researchers and practitioners interested in process intensification and will also help in further research, developments, and industrial applications of process intensification.

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