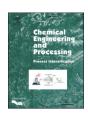
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## Process intensification control: Advancing efficiency and sustainability, a review

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#### ABSTRACT

Process intensification has revolutionized chemical process design by integrating reaction and separation, enhancing efficiency, reducing energy consumption, and promoting sustainability. However, these advancements introduce significant control challenges due to increased process complexity, nonlinear interactions, and dynamic constraints. Over the past 25 years, conventional control strategies have been progressively replaced by predictive, adaptive, and data-driven methods, which are better suited for managing multivariable interactions and real-time optimization. The widespread adoption of predictive control frameworks has improved stability, reduced response times, and enhanced energy efficiency in reactive and extractive distillation, dividing-wall columns, and hybrid separation processes. Furthermore, integrating intelligent decision-making tools has enabled real-time adaptability, ensuring robust performance under fluctuating operating conditions. The emergence of hybrid control strategies, which combine predictive models with data-driven learning techniques, has further enhanced the ability to address nonlinearities and process uncertainties. This shift underscores a transition toward more intelligent and sustainable process operations, where control systems not only optimize efficiency but also minimize emissions and improve resource utilization. As process intensification continues to advance, future research should focus on scalable, autonomous, and computationally efficient control solutions to ensure operational reliability and economic feasibility in sustainable chemical manufacturing.

Nomencle	ature
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AI artificial intelligence
ANN artificial neural networks
ARMA autoregressive moving average

ATJ alcohol-to-jet

BTX benzene-toluene-xylene CN condition number

CSTR continuous stirred-tank reactor DAE differential-algebraic equation

DES deep eutectic solvents
DMC dimethyl carbonate
DMCo dynamic matrix control

EDWCs extractive dividing-wall columns

EG ethylene glycol

EMPC economic model predictive control

GOBF generalized orthonormal basis filter
HIDiC heat-integrated distillation columns

IAE absolute error

ICS intelligent control system
LCA life cycle assessment
DWC dividing wall column
MEC micro-engineered catalyst

MIDO mixed-integer dynamic optimization
MINLP mixed-integer nonlinear programming
mp-MPC multi-parametric model predictive control

MRI morari resiliency index MSE mean squared error NLP nonlinear programming

NMPC nonlinear model predictive control

PI process intensification

PID proportional-integral-derivative controller

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P-I proportional-integral controller MPC model predictive control

PMF photocatalysis-membrane filtration

PMRs photocatalytic membrane reactors

PRI process route index
PSD pressure-swing distillation
PSO particle swarm optimization
QDMC quadratic dynamic matrix control
R-DWC reactive divided wall columns

RGA relative gain array

RSM response surface methodology

SMC sliding mode control

SVD singular value decomposition

TAC total annual cost

TCED triple-column extractive distillation

TOC total organic carbon
TPA terephthalic acid

#### 1. Introduction

Process intensification (PI) has redefined chemical and process engineering, offering solutions to the pressing demands for efficiency, sustainability, and resilience in industrial systems [1]. By integrating unit operations and optimizing resource utilization, PI achieves higher productivity while significantly reducing energy consumption, emissions, and waste [2]. Its applications across diverse industries—pharmaceuticals, petrochemicals, and energy—demonstrate its potential to transform traditional manufacturing practices into streamlined, cost-effective, and environmentally friendly systems [3]. However, the success of PI heavily depends on the design and implementation of robust control systems capable of managing its inherent complexity and dynamic behavior [4,5].

PI was initially considered challenging in terms of control due to its complex topology and the integration of multiple unit operations within a single unit. This led to the assumption that intensified processes would inherently exhibit inferior controllability compared to conventional systems. However, subsequent research has demonstrated that this is not necessarily the case, as several studies have shown that control properties can be comparable or even superior to those of traditional processes. For example, Agrawal et al. [6] initially identified the control complexity of the Petlyuk column; however, later findings by Alvarez-Ramirez & Monroy-Loperena [7] suggested that a simple Proportional-Integral (P-I) controller could be sufficient for effective operation. Moreover, Skogestad [8] emphasized that control entails selecting degrees of freedom, which fundamentally shapes control strategy design. This adds another layer of complexity, making control design in intensified processes non-trivial yet not inherently unmanageable.

Traditional control methods, including Proportional-Integral-Derivative (PID) and P-I controllers, have long been the cornerstone of process automation [9]. These methods are favored for their simplicity, reliability, and effectiveness in maintaining steady-state operations across a wide range of applications [10]. PID controllers, for example, excel in controlling processes with relatively stable and predictable dynamics, offering precise setpoint tracking and disturbance rejection. However, when applied to intensified processes, traditional control systems often encounter significant limitations due to the nonlinear, multi-scale, and highly dynamic nature of these systems [11]. These constraints necessitate a shift toward advanced control strategies capable of addressing the unique challenges posed by P-I.

Modern advancements in control systems have introduced innovative techniques, such as Model Predictive Control (MPC), which provide predictive capabilities and handle multivariable interactions with operational constraints. MPC has proven particularly effective in intensified systems like reactive distillation and membrane separations, where dynamic interactions and rapid response requirements are critical

[12]. Moreover, hybrid control systems that integrate traditional PID controllers with artificial intelligence (AI) models have emerged as promising solutions [13]. These hybrid systems combine the robustness of traditional control with the adaptability and learning capabilities of AI, enabling real-time optimization and fault detection in complex processes [14].

The emergence of digital twins has further revolutionized the control landscape for PI [15]. Digital twins are virtual replicas of physical processes that allow real-time simulation, monitoring, and optimization of operations [16]. These tools have gained traction for their ability to enhance decision-making by providing predictive insights and enabling proactive adjustments to maintain optimal performance. For instance, digital twin technology has been applied in intensified separation processes to dynamically simulate and adjust operational parameters, improving energy efficiency and reducing downtime [17].

Sustainability is at the core of PI, driving innovation in control systems to meet environmental and operational goals [1]. Traditional PID and P-I controllers, while effective in steady-state scenarios, lack the flexibility to accommodate rapid changes in operating conditions and environmental constraints. Advanced control systems incorporating AI and machine learning algorithms have demonstrated their potential to significantly reduce energy consumption, improve resource utilization, and minimize waste [18]. For instance, Ukoba et al. [19] emphasized how advanced AI-based control strategies optimize the monitoring and operation of renewable energy systems, enhancing grid stability and flexibility through precise forecasting techniques and real-time dynamic adjustments. Similarly, circular economy principles have been integrated into PI systems, promoting resource recovery and by-product reuse through centralized control platforms [20].

Despite these advancements, challenges remain in scaling advanced control strategies to industrial applications. Many industries still rely heavily on legacy PID-based systems, making the transition to AI-enhanced or MPC frameworks resource-intensive and complex. Furthermore, intensified processes often require sophisticated sensor networks and high-fidelity data acquisition systems to enable real-time monitoring and control [21]. Scalability, interoperability, and cost-effectiveness are critical barriers to the widespread adoption of these technologies.

This review provides a comprehensive analysis of the state-of-the-art in process intensification control, examining the evolution from traditional PID and P-I controllers to advanced AI-driven and predictive strategies. It explores the integration of digital twins and sustainability-focused innovations, highlighting their transformative impact on operational efficiency and environmental performance. The primary contribution of this work lies in bridging foundational control techniques with cutting-edge technologies, offering a holistic perspective that identifies challenges, opportunities, and future research directions in the field of process intensification control.

#### 2. Overview of process intensification

PI represents a transformative approach in chemical engineering, aiming to enhance the efficiency, sustainability, and compactness of industrial processes [22]. By integrating multiple operations, optimizing energy and material utilization, and minimizing equipment size, PI addresses the growing demand for environmentally and economically viable production methods [23]. Among the various technologies developed under the PI framework, intensified distillation processes stand out for their ability to tackle energy-intensive separation challenges, making them a focus of this review.

PI encompasses a diverse range of technologies designed to optimize energy and resource usage while minimizing operational complexity (Table 1). These include microreactors, which enable precise reaction control in compact systems; compact heat exchangers, which enhance thermal efficiency; plasma-assisted processes, which use plasma energy to accelerate chemical reactions; and supercritical fluid technologies,

**Table 1**Key intensified processes and their characteristics.

Intensified Process	Description	Key Applications	Benefits
Dividing Wall Columns (DWCs)	A single column with an internal dividing wall to achieve multiple separations; thermodynamically equivalent to Petlyuk configurations.	Petrochemicals, alcohol separation.	Reduced energy consumption and equipment footprint.
Extractive Distillation	Uses a solvent to alter the relative volatilities of components for separation.	Purification of azeotropic or closeboiling mixtures.	Higher purity with lower energy requirements.
Reactive Distillation	Integrates chemical reaction and distillation in a single column.	Production of esters, biodiesel, and specialty chemicals.	Reduces equipment size and enhances reaction efficiency.
Membrane- Assisted Distillation	Combines membrane technology with distillation for enhanced separation.	Water purification, alcohol recovery, and hybrid separations.	Improved selectivity and lower operational costs.
Microreactors	Compact systems that enable precise control over reaction kinetics and thermodynamics.	Pharmaceuticals, fine chemicals.	Faster reaction rates, scalability, and reduced waste.

which leverage the unique properties of supercritical states for efficient extraction and reaction [24].

The distillation technologies—dividing wall columns (DWCs), Extractive Distillation (ED), reactive distillation, and membrane-assisted distillation—exemplify the principles of PI by integrating or enhancing traditional separation methods to achieve superior performance [25]. DWCs, for instance, integrate multiple separation steps into a single unit, significantly reducing energy consumption and equipment size [26]. It is important to note that DWCs are thermodynamically equivalent to Petlyuk configurations, which provide an alternative design approach depending on process constraints and design preferences [27].

ED introduces a selective solvent to alter the relative volatilities of components, enabling the separation of azeotropic or closely boiling mixtures [28]. This method is widely used in the purification of solvents, specialty chemicals, and fuels [29]. Reactive distillation, on the other hand, integrates reaction and separation in a single unit, optimizing equilibrium-limited reactions such as esterification and biodiesel production [30]. Finally, membrane-assisted distillation combines the selectivity of membranes with the separation capabilities of distillation, enhancing energy efficiency and broadening the applicability of traditional methods [31].

While there are numerous intensified processes within the broader scope of PI, the focus on these distillation technologies reflects their widespread industrial relevance and their transformative potential in separation processes. This targeted approach enables an in-depth exploration of their principles, applications, and contributions to advancing industrial efficiency and sustainability.

#### 3. Challenges in control of intensified processes

The control of intensified processes represents a paradigmatic challenge that transcends conventional techniques due to the convergence of nonlinear dynamics, extreme physical constraints, and the need for unprecedented operational integration [32]. As the chemical and process industries transition toward more compact, sustainable, and highly efficient configurations, complex issues arise related to operational stability, the predictive capability of models, and the adaptability of control systems to abrupt changes in system conditions.

These limitations are further exacerbated in intensified processes,

where dynamic interactions, low residence times, and reduced process inertia lead to heightened sensitivity to disturbances. Variability in feedstock composition, sensor noise, actuator saturation, and modelplant mismatch are all examples of process uncertainties that can destabilize performance when not properly managed. Therefore, successful control strategies must be designed with built-in resilience to such disturbances, particularly in multiscale systems characterized by tight coupling and limited buffering capacity.

One of the most intriguing aspects in this field is the difficulty of modeling emergent behaviors arising from interactions between coupled processes. For instance, in multifunctional reactors where reaction and separation occur simultaneously, small fluctuations in one variable can unpredictably amplify, driving the system into undesirable operating zones [11]. This high sensitivity necessitates control systems that are not only robust but also capable of anticipating nonlinear phenomena through advanced learning algorithms and simulations.

Additionally, the use of distributed sensors and monitoring systems based on big data creates a scenario where the volume of information generated surpasses the processing capacity of many traditional tools [33]. Advances in hardware, particularly Graphics Processing Units (GPUs), play a pivotal role in addressing these challenges. GPUs provide massively parallel computing capabilities, enabling the processing and analysis of substantial volumes of data in real-time [34]. This advancement facilitates the implementation of predictive control systems based on hybrid models, which integrate real-time data with simulations grounded in physical principles, ensuring efficient management of computational demands.

Technologies such as MPC based on hybrid models—combining realtime data with simulations grounded in physical principles—offer a promising avenue, albeit one still underutilized due to computational and methodological limitations [35–37]. These solutions must be capable of identifying critical patterns in real-time, enabling proactive rather than reactive control decisions.

Furthermore, intensified processes often operate under extreme regimes of pressure, temperature, and reaction rates, imposing severe constraints on material selection and the design of sensors capable of enduring such conditions without compromising precision or durability. The lack of adaptive control tools for these extreme environments limits the industrial application of these technologies, emphasizing the need for interdisciplinary collaborations among materials engineering, informatics, and control engineering [38].

Finally, scaling these solutions from pilot systems to industrial environments remains a critical obstacle. Discrepancies between models developed in laboratories and the inherent complexities of industrial operations, such as interactions with external systems and economic constraints, hinder the direct application of many advanced control strategies [39]. This underscores the importance of scalable and adaptive approaches that account for both uncertainty and the variability intrinsic to industrial processes.

The control of intensified processes demands a paradigm shift integrating hybrid modeling tools, artificial intelligence, and material design to address the challenges of stability, predictability, and scalability. The convergence of these approaches can pave the way for a future where process intensification becomes not only a technological reality but also an industrial standard.

#### 4. Recent advances in process intensification control

This review analyzes 118 peer-reviewed articles published between 2000 and 2025, retrieved from the Scopus database. The selection process was based on the following keywords: "Process Intensification Control," "Advanced Control Strategies," "Reactive Distillation Control," "Dividing Wall Column Control," and "Extractive Distillation Control." The reviewed articles were classified into two primary categories: (1) the type of intensified process and (2) the control techniques employed. The classification framework encompasses reactive separation systems

(reactive distillation, extractive distillation, and dividing wall columns), catalytic and membrane reactors, hybrid separation processes, and energy-integrated distillation. Furthermore, control strategies were categorized into classical control (PID-based methods), advanced control (MPC, NMPC, ANN, soft sensors), and hybrid techniques integrating AI and model-based control.

#### 4.1. Classification of intensified processes and control strategies

The reviewed works were grouped according to the intensified process they addressed and the control strategies implemented. Table 2 summarizes the classification and the primary findings.

#### 4.2. Key findings and quantitative insights

The evaluation of control strategies in process intensification highlights key findings across different intensified processes. Each technology, including RD, ED, DWC, Hybrid Membrane-Reactors, and Catalytic Reactive Systems, has adopted specific control techniques to enhance efficiency and stability. The following section presents quantitative insights into how these processes benefit from advanced control approaches, improving performance, energy savings, and overall operational effectiveness.

#### 4.2.1. Reactive distillation (RD)

In this section, the main works related to the control of RD columns are presented, highlighting advances in control strategies, operational optimization, and improvements in the stability and efficiency of these intensified systems.

Al-Arfaj and Luyben [40] analyzed control strategies for an ideal two-product RD column, evaluating six alternative structures. They found that integrating composition analyzers in the reactive zone improved reactant inventory control and reaction stoichiometry. Dynamic simulations showed that increasing reactive zone holdup (1 to 2 kmol/tray) significantly enhanced controllability, maintaining product purities of up to 98 % under a 20 % feed flow disturbance. Single-end temperature control stabilized product quality while simplifying control loop interactions. Overdesigning the reactive section (increasing catalyst holdup or adding reactive trays) improved both steady-state and dynamic control. Feedback-based feed adjustments prevented stoichiometric imbalances, ensuring stability under varying conditions.

Balasubramhanya and Doyle [41] developed a reduced-order nonlinear model for nonlinear model-based control (NMPC) of batch RD columns, reducing computational complexity while maintaining accuracy. The model, based on traveling wave phenomena, required only five differential and six algebraic equations instead of the original 31 differential equations. Applied within an NMPC framework, it achieved tight distillate composition control through tray temperature regulation. The approach reduced simulation time by a factor of 6.5 while maintaining comparable control performance, demonstrating the potential of reduced-order models for efficient NMPC in RD systems.

Vora and Daoutidis [42] investigated control strategies for an RD column producing ethyl acetate, implementing a multiple-feed configuration to enhance conversion (from 66% to 76.8%) and product purity (from 54% to 65%). They identified a two-time-scale dynamic behavior, where conventional multiloop controllers struggled with input

**Table 2**Control techniques applied in process intensification technologies.

<b>Process Intensification Technology</b>	<b>Control Techniques Used</b>
Reactive Distillation (RD)	PID, MPC, NMPC, ANN, Soft Sensors
Extractive Distillation (ED)	PID, MPC, Dual Temperature Control
Dividing Wall Columns (DWC)	MPC, Inferential Control, Ratio Control
Hybrid Membrane-Reactors	MPC, ANN, Self-Optimizing Control
Catalytic Reactive Systems	NMPC, Adaptive MPC, AI-based Control

multiplicity and nonlinearities. A nonlinear feedback controller based on an exact dynamic model outperformed SISO P-I controllers, improving setpoint tracking and disturbance rejection. A control strategy focusing on slow dynamics enhanced robustness against modeling errors, reducing instability risks, and highlighting the necessity of nonlinear control in RD systems.

Al-Arfaj and Luyben [43] examined control strategies for an olefin metathesis RD column, evaluating three steady-state designs with varying pressure and conversion levels. Control studies demonstrated that dual temperature control—manipulating tray temperatures via reflux and boilup rates—maintained product purity under  $\pm 25~\%$  feed disturbances. Higher pressure designs with additional trays reduced product quality deviations. The study concluded that temperature-based control structures detect disturbances faster than composition-based control, providing an optimal balance of economic efficiency and operability.

Georgiadis et al. [44] explored the integration of design and control in RD systems for ethyl acetate production, comparing sequential and simultaneous optimization approaches. The simultaneous method reduced annual costs by 5 % (\$220,000) while improving controllability. Optimal design parameters, including a column diameter of 6.37 m and heat exchanger areas of 315 m² (reboiler) and 425 m² (condenser), enhanced dynamic performance. The system managed sinusoidal feed disturbances and diurnal cooling variations, achieving tighter bottom product purity control and a 10 % reduction in integral square error. Their findings demonstrated the advantages of integrating design and control using mixed-integer dynamic optimization (MIDO) for process intensification.

Al-Arfaj and Luyben [45] conducted an in-depth control study on methyl acetate RD, evaluating three control structures—CS1, CS5, and CS7—under high- and low-conversion scenarios. CS1, which used three composition controllers for stoichiometric feed control, struggled with nonlinearity in high-conversion conditions but achieved 95 % conversion and 95 % purity in low-conversion cases. CS5, integrating a composition controller with a stripping section temperature controller, maintained methyl acetate purity between 95.98 % and 96.03 % and water purity above 98.55 %, even under 20 % acetic acid feed disturbances. CS7, a temperature-based control structure optimized using singular value decomposition (SVD), identified the most sensitive trays and maintained product purities close to specification despite a 20 % reboiler duty increase. The findings highlight the superiority of temperature-based control in handling nonlinearities and improving robustness under high-purity operations.

Al-Arfaj and Luyben [46] analyzed the control of ethyl tert-butyl ether (ETBE) RD columns, comparing double-feed and single-feed systems. The double-feed configuration required internal composition control to balance stoichiometry, whereas temperature control alone sufficed for the single-feed setup under moderate disturbances. The optimized double-feed system, producing 700 kmol/h of ETBE with 99 % conversion, showed superior dynamic performance when butene feed manipulation was used for internal composition control, achieving 99 % ETBE purity in the bottoms with minimal ethanol losses. Simulations demonstrated that direct composition control provided better robustness against feed rate and composition disturbances (+25 % and  $\pm 10$ %), whereas temperature control alone risked purity losses under larger disturbances.

Grüner et al. [47] developed a nonlinear control strategy for RD columns using input/output-linearization combined with an observer, achieving enhanced performance in industrial-scale applications. The controller, relying solely on temperature measurements, maintained product purity under  $\pm 5\,\%$  feed composition disturbances. Compared to a well-tuned linear controller, the nonlinear approach reduced settling times, achieving faster convergence with a 75 K setpoint change and improved decoupling of tray temperatures (T4 and T60). Additionally, it provided superior disturbance rejection, stabilizing key compositions under  $\pm 10\,\%$  feed flow variations, demonstrating the advantages of

nonlinear control for operational robustness in intensified processes.

Al-Arfaj and Luyben [48] studied the plantwide control of TAME RD production, identifying the RD column as the critical unit. Their control strategy, combining methanol feed regulation with temperature control on specific trays, ensured stable operation under  $\pm 20\,$ % feed disturbances. The optimized design achieved 92 % isoamylenes conversion with minimal methanol consumption (508.31 kmol/h), maintaining separation efficiency with low reflux ratios (0.5 for methanol and 1 for C5s). These results confirm that integrating process-wide control structures enhances stability and efficiency in etherification RD systems.

Huang et al. [49] proposed a temperature control framework for heterogeneous RD processes, addressing vapor-liquid-liquid equilibria (VLLE) and kinetically controlled reactions. Their design, including optimized feed tray locations and decentralized PI controllers, achieved 99 % product purity while maintaining low acid impurity levels. Using the nonsquare relative gain (NRG) method, they identified optimal temperature-control trays, enabling one-way decoupled multivariable control. Feedforward temperature compensation eliminated steady-state offsets and improved transient responses, ensuring stringent purity specifications under production rate variations. The results underscore the potential of temperature-based control for enhancing the stability and efficiency of heterogeneous RD systems.

Kaymak and Luyben [50] conducted a comparative study of two temperature-based control structures for RD columns, evaluating their dynamic performance under disturbances. CS7, using two P-I controllers to regulate fresh feed flow rates, showed rapid stabilization but aggressive responses. In contrast, CS8 combined feed and reboiler heat duty manipulations, improving sensitivity and dynamic stability through optimal tray pairings. In the methyl acetate case, CS8A outperformed CS8B, maintaining product purity within 1 % of the 95 % target under disturbances, while CS8A effectively controlled feed composition variations up to 5 %. The study highlighted the importance of sequential tuning in interacting controllers to enhance system stability in complex RD processes.

Khaledi and Young [51] developed a 2  $\times$  2 unconstrained MPC scheme for controlling product purity and reactant conversion in an ETBE RD column, addressing nonlinearities and process gain bidirectionality. Using a first-order plus dead time model, their MPC controller achieved robust disturbance rejection and smooth set-point tracking. At 100 kmol/h feed flow and 950 kPa operating pressure, a+3 °C step change at stage 7 demonstrated superior performance over PI controllers, maintaining isobutylene conversion above 98 % and ETBE purity at 88.7 wt %. The results underscored the effectiveness of MPC in stabilizing complex RD systems despite feed composition variations and measurement noise.

Olanrewaju and Al-Arfaj [52] proposed a linearized state-space model for RD process control, addressing delays in online composition analyzers by implementing a Kalman filter-based state estimator. The estimator, coupled with a dual-end composition control strategy, maintained setpoints within  $\pm 2$ % despite plant-model mismatches and disturbances. When the reactant B feed flow increased by 10 %, doubling the controller gains improved response times and stability. The study highlighted that while small estimation errors were tolerable, significant deviations in volatilities or initial conditions degraded control accuracy, demonstrating the viability of linear state estimators in enhancing operational efficiency in RD processes.

Panjwani et al. [53] developed a mixed-integer dynamic optimization (MIDO) framework for simultaneous RD system design and control, achieving a 17 % reduction in total annualized costs. The optimal configuration, integrating column diameter, tray configurations, and reboiler/condenser surface areas, reduced reflux rate and steam consumption by 8 % and 5 %, respectively. Their novel control scheme, manipulating steam flow for feed tray temperature regulation rather than direct composition control, demonstrated improved operability under acetic acid inlet composition and cooling water temperature disturbances. The study reinforced the advantages of integrating design

and control to optimize intensified processes, with potential applications in MTBE and ETBE production.

Hung et al. [54] analyzed the control of RD systems for the esterification of acetic acid with C1–C5 alcohols, evaluating three process configurations using nonlinearity indices and sign reversal fractions. Their findings showed that BuAc exhibited minimal nonlinearity and high stability, while MeAc faced severe dynamic challenges due to input multiplicities. Temperature control strategies in decentralized configurations resulted in settling times of approximately 5 h for BuAc and AmAc, and up to 15 h for MeAc. TAC values ranged from \$482.54k for BuAc to \$1.04 M for MeAc at production rates of 52,825 tons/year. The study emphasized the critical role of process-specific control strategies in improving RD system performance and stability.

Kawathekar and Riggs [55] investigated the application of NLMPC to an ethyl acetate RD column, demonstrating its superiority over P-I controllers in managing strong nonlinearities. For the [L/D,V] configuration, NLMPC reduced the Integral of Absolute Error (IAE) from 2.41 to 0.91 for the overhead loop and from 6.84 to 2.14 for the bottom loop, representing a 2–3-fold improvement in control performance. NLMPC also maintained stability under unmeasured disturbances and exhibited resilience to process-model mismatches of up to 25 %. Additionally, implementing a two-column configuration with a recovery column enabled high-purity ethyl acetate production (99.5 %), highlighting the advantages of NLMPC in process intensification through enhanced disturbance rejection and dynamic performance.

Lee et al. [56] developed advanced control strategies for ethyl acetate RD, optimizing sensor placement via closed-loop sensitivity analysis. Their dual-point control strategy maintained EtAc purity above 99.5 % under  $\pm 20$  % throughput variations, reducing impurity deviations to 2 %. Sensor relocation improved steady-state performance and dynamic controllability, mitigating overshoot and deviations caused by acid feed composition fluctuations. The study quantified trade-offs, showing that while dual-point control improved operability and disturbance rejection, it introduced more oscillatory responses than single-point control. These findings underscore the role of sensor optimization in enhancing stability and efficiency in RD processes.

Kumar and Kaistha [57] analyzed the impact of steady-state multiplicities on methyl acetate RD control, demonstrating that fixed reflux rate policies induced unwanted transitions, while a fixed reflux ratio approach improved stability. A novel rangeability metric quantifies input multiplicity severity, guiding the selection of optimal control variables. Dynamic simulations showed that controlling a pseudo-output ( $\Delta T = T20\,$  - T8) enhanced robustness, whereas controlling the most sensitive tray temperature (T18) led to instability. Ratio control between feeds and reboiler duty enabled the system to handle production rate increases of up to 40 %, highlighting the necessity of systematic sensitivity analysis for robust RD control.

Kumar and Kaistha [58] examined two RD configurations—5–10–5 (10 reactive trays) and 5–20–5 (20 reactive trays)—to assess their dynamic behavior and control performance. The 5–20–5 design handled  $\pm 20$ % throughput disturbances within 4 h, whereas the 5–10–5 design exhibited slower responses. CS2, which manipulated the heavy reactant feed, outperformed CS1 in transient stoichiometric balance, stabilizing throughput changes up to  $\pm 70$ %. Controlling reactive tray temperature (T15) provided superior disturbance rejection compared to rectifying tray control (T18). Despite an 11.8 % increase in vapor boil-up and slightly higher cost (\$310,730/year vs. \$298,160/year), the 5–20–5 configuration demonstrated better sensitivity and controllability, emphasizing the importance of optimized catalyst distribution and tray design.

Hsu et al. [59] proposed an intensified RD and ED process for dimethyl carbonate (DMC) and ethylene glycol (EG) production, achieving complete ethylene carbonate (EC) conversion with excess methanol. Using aniline as an entrainer, they enhanced methanol-DMC relative volatility, reducing reboiler duty by 32.8 %, entrainer feed ratio from 1.965 to 0.883, and column heights, cutting ED stages from 48 to

32 and recovery column stages from 32 to 18. Simple tray temperature control loops maintained 99.5% DMC and 99.99% EG purities, ensuring stability under feed and throughput variations. These results demonstrated the economic and operational advantages of the aniline-based separation strategy in RD systems.

Wang et al. [60] developed an integrated plant-wide control framework for DMC and EG production via RD coupled with thermally coupled ED, achieving a 17.6 % reduction in reboiler duty compared to conventional ED. The control strategy employed steady-state analysis to optimize manipulated and controlled variables, ensuring product purities of 99.8 % DMC and 99.99 % methanol. Temperature control loops effectively mitigated disturbances, maintaining stoichiometric balance under feed rate fluctuations and vapor split ratio variations. Dynamic simulations confirmed that the control scheme maintained process deviations within acceptable margins, establishing thermally coupled ED as an energy-efficient strategy for RD systems.

Kim et al. [61] applied nonlinear wave propagation theory to the control of an RD column for terephthalic acid (TPA) synthesis, optimizing composition profile positioning to enhance conversion and purity. A wave propagation model demonstrated that continuous methanol removal minimized equilibrium limitations, enabling near-complete conversion. Profile position control, adjusting vapor and liquid flow rates, stabilized wave positions, and maintaining TPA yield above 98 % with minimal methanol contamination. The control framework outperformed traditional temperature control, ensuring fast recovery from  $\pm 20$ % feed flow variations, underscoring the potential of nonlinear wave theory in process intensification.

Sharma and Singh [62] reviewed advanced RD control strategies, highlighting MPC, DMC, QDMC, and NMPC as superior alternatives to conventional PI/PID controllers. Case studies demonstrated NMPC's improved composition control in ethyl acetate RD, dual-temperature control's robustness in methyl acetate RD, and composition control's effectiveness in MTBE decomposition. Additionally, RD integrated with DWC achieved 50 % energy savings. Dual-temperature loops in RDWDC enhanced ethyl acetate synthesis stability under disturbances. These findings reinforce the role of advanced control strategies in optimizing RD operability and efficiency, setting the stage for AI-driven adaptive control in intensified systems.

Lin et al. [63] optimized reactive section distribution in an olefin metathesis RD column, comparing four configurations. Design-II reduced reboiler duty by 5.27 % but degraded controllability, while Designs III and IV, which extended the reactive section, achieved reboiler duty reductions of 4.51 % and 4.30 %, maintaining superior control performance. A dual-point temperature control scheme showed that Design-II had larger bottom purity deviations, whereas Designs III and IV minimized steady-state errors. Control performance metrics, including IAE and steady-state deviation (SSD), confirmed that reactive section distribution significantly influences both energy efficiency and dynamic stability in RD systems.

Nikacevic et al. [64] analyzed control challenges in intensified processes such as RD, DWC, and micro-scale reactors, emphasizing NMPC's superiority over traditional controllers. In an ETBE RD column, a  $2\times 2$  unconstrained NMPC scheme reduced composition variability by 35 % and improved conversion efficiency by 25 % compared to PID control. The study highlighted NMPC's robustness against nonlinearities and process interactions, reinforcing its potential for industrial-scale applications in process intensification.

Ignat and Kiss [65] designed an R-DWC for FAME production, integrating RD and DWC technologies to achieve 39 % fewer stages, 57 % fewer reactive trays, and only a 1.5 % heat duty increase. By feeding alcohol as vapor, the system improved product purity and impurity control in side streams. SVD identified sensitive trays for inferential temperature control, enabling robust disturbance rejection, including production rate fluctuations and catalyst deactivation. The control scheme maintained 99.8 % purity for methanol and water, demonstrating R-DWC's potential for optimizing energy efficiency and

sustainability in biodiesel production.

Seban et al. [66] developed an MPC framework for RD columns, integrating Generalized Orthonormal Basis Filter (GOBF) and Autoregressive Moving Average (ARMA) models to enhance dynamic process representation. The GOBF-ARMA MPC achieved superior control performance, precisely tracking a distillate purity setpoint increase from 0.95 to 0.96 mol % while minimizing energy consumption. A 2.5 % feed rate disturbance was effectively mitigated, demonstrating robust disturbance rejection. This approach optimizes energy use through *in situ* heat integration, improving operational reliability and safety, and highlighting its potential for broader industrial applications.

Segovia-Hernández et al. [67] reviewed RD control advancements, focusing on deterministic and stochastic optimization methods to enhance process intensification. Deterministic approaches, such as MINLP and dynamic programming, optimized design parameters, achieving over 20 % energy savings. Stochastic techniques, including NSGA-II, improved multi-objective RD optimization, enabling 99.9 % purity in esterification processes while reducing energy consumption and  $\rm CO_2$  emissions by 25 %. Case studies on methyl acetate and ETBE demonstrated substantial cost reductions and improved control performance, emphasizing the role of simultaneous design and control optimization in RD sustainability.

Valluru et al. [68] introduced a real-time optimization (RTO) and adaptive NMPC framework for RD systems, integrating a nonlinear Bayesian estimator (DAE-EKF) to update models dynamically. Applied to an RD column with reaction  $A+B\rightleftharpoons C+D$ , this approach maintained product concentration deviations below 1 % under a 10 % reactant B feed rate disturbance. The adaptive NMPC, with prediction and control horizons of 40 and 4, respectively, ensured offset-free mole fraction control. Despite 20 min RTO computation times, the strategy dynamically optimized product qualities, proving its economic and operational benefits for intensified RD systems.

Baldea [69] analyzed the impact of process intensification on control dynamics, demonstrating that high material recycling rates reduce equipment but accelerate responses. size system reaction-separation-recycle RD systems, intensified configurations with vapor holdups of 1338 mol exhibited nearly twice the response speed of integrated systems (1404 mol), as confirmed by eigenvalue analysis. However, the faster dynamics introduced tighter process coupling and control complexities. The study emphasized that intensified systems require real-time MPC to handle nonlinear interactions, underscoring process intensification's unique control challenges and efficiency opportunities.

Mansouri et al. [70] integrated process design and control in RD systems using reactive driving force diagrams to optimize controllability. Dynamic simulations showed that operating at maximum driving force improved stability, with a 12 % isobutene feed step increase resulting in stable product compositions and minimal reflux ratio and reboiler duty adjustments. Relative Gain Array (RGA) analysis confirmed minimized loop interactions, facilitating robust controller design. This methodology highlights the potential for achieving resilient and sustainable RD systems through integrated design and control.

Mansouri et al. [71] proposed a computer-aided framework for simultaneous RD process design and control, applying Mixed-Integer Dynamic Optimization (MIDO) for economic feasibility and control performance. In a case study on MTBE synthesis, the optimized design achieved 98 % isobutene recovery and 84 % MTBE purity, with a methanol conversion of 83.15 %. Step-response analysis and RGA calculations verified that reflux-to-distillate and reboiler-to-bottom product control pairings minimized disturbances while maximizing controllability. This integrated approach enhances energy efficiency and sustainability, demonstrating the benefits of process intensification control.

Ramírez-Márquez et al. [72] evaluated control strategies for a multitasking RD column producing high-purity silane, dichlorosilane, and monochlorosilane. Under 10 % feed flowrate disturbances and 5 %

contamination, temperature control stabilized the process with short settling times ( $\sim$ 3–5 h), despite minor steady-state deviations. Composition control achieved faster responses ( $\sim$ 2–5 h) but required online chromatographic measurements, limiting industrial feasibility. The cascade strategy provided the best disturbance rejection and eliminated steady-state offsets but had longer settling times ( $\sim$ 7–10 h) and increased complexity. Optimal column pressure (2.3 atm) balanced transient response and energy efficiency, enabling seamless transitions between product outputs by adjusting reflux ratio, reboiler duty, and column pressure, ensuring flexible and stable high-purity silane production.

Maya-Yescas et al. [73] analyzed process intensification control, focusing on stability and manipulated variable selection. In an intensified RD biodiesel process, energy integration reduced cooling demand by 91.8 % and heating requirements by 77.8 %, though it introduced control challenges due to the loss of degrees of freedom. Advanced control methods, such as nonlinear observers and Kalman filtering, improved disturbance rejection. In FCC operations, improper variable pairings led to unstable control despite multiple manipulated variables, highlighting the need for robust observability analysis and optimal control pairings to ensure process stability in highly intensified systems.

Mahindrakar and Hahn [74] implemented MPC for RD benzene hydrogenation, addressing process nonlinearity and fluctuating benzene concentrations (3–11 vol %). SISO MPC with input disturbance correction outperformed P-I control, reducing benzene concentration deviations by 65 % and shortening settling times from 219 to 127 min. MIMO MPC showed no additional benefits due to weak variable interactions, confirmed by RGA analysis. The results demonstrated that incorporating an input disturbance model enhances rejection performance without requiring real-time composition measurements, offering a cost-effective solution for intensified RD systems.

Chen et al. [75] developed a thermally coupled RD system for methyl valerate (VAME) production, achieving a 30.3 % energy reduction, though TAC decreased only by 17 % due to compressor inclusion. Dynamic simulations under  $\pm 20$  % throughput and  $\pm 5$  % composition disturbances showed faster stabilization and smaller steady-state deviations compared to conventional setups. Product purities (99 mol % VAME and water) remained stable, confirming the thermally coupled system's robustness. The study highlights thermal coupling as a viable strategy for enhancing both economic and operational performance in industrial-scale RD applications.

Mansouri et al. [76] introduced a hierarchical decomposition framework integrating process design and control for multi-element RD systems. In MTBE production, the optimal seven-stage RD design required 856.6 kW, significantly lower than alternative designs exceeding 2000 kW. Dynamic performance analysis demonstrated rapid rejection of  $a+16.5\,\%$  methanol feed disturbance with minimal overshoot. Sensitivity analysis, RGA, and MPC outperformed P-I controllers, reducing control effort and enhancing disturbance rejection. The study underscores how integrating process intensification control optimizes energy efficiency, economic viability, and operational robustness in chemical processes.

Giwa et al. [77] applied MPC to a biodiesel RD system, optimizing tuning parameters (control horizon: 11, prediction horizon: 18, manipulated variable rate weight: 0.05, output weight: 2.17) to achieve set-point tracking within 60 min with minimal oscillations. Under a 0.4-unit step change, IAE and ISE were reduced to 6.05 and 2.05, respectively, demonstrating MPC's efficiency in servo control. However, disturbance rejection exhibited prolonged settling times (~800 min), indicating challenges in dynamic disturbance management. Compared to PID control, MPC provided superior precision and reduced oscillations, though further tuning is needed for improved response under variable operating conditions.

Dias and Ierapetritou [78] reviewed intensified process control advancements, highlighting NMPC's capability to handle dynamic disturbances but noting computational complexity as a limitation.

Multi-parametric NMPC (mp-MPC) emerged as a viable alternative, reducing online computation times in pressure swing adsorption. Integrating scheduling with control enhanced transient operation efficiency, while EMPC outperformed conventional strategies in economic performance. The study emphasized the potential of AI and parallel computing in real-time optimization, improving sustainability and efficiency in chemical process industries.

Ge et al. [79] optimized RD and RDWC configurations for formic acid production using genetic algorithms, with RDWC achieving a higher methyl formate conversion (88.7 %) than RD (72.4 %) despite a slight increase in energy consumption (8.2 %) and total annual cost (4.3 %). Dynamic control comparisons showed that MPC significantly outperformed PI control, reducing maximum deviations and improving settling time and ISE. For  $a\pm10$  % feed disturbance, MPC reduced tray temperature deviations (e.g., T40, RD) from 8.4 °C under PI to 1.3 °C, demonstrating its effectiveness in managing nonlinear interactions and improving system operability.

Sakhre [80] conducted a comprehensive review of advancements in RD control, focusing on strategies to address nonlinearity, process efficiency, and optimization techniques. The study explored the application of MINLP for RD configuration optimization, highlighting its role in achieving cost-effective designs and improving process feasibility. The review emphasized the importance of integrating model-based control approaches to enhance stability, minimize energy consumption, and ensure robust operation in intensified RD systems. These findings demonstrate the ongoing evolution of control methodologies aimed at increasing efficiency and sustainability in reactive distillation.

Pistikopoulos et al. [81] analyzed intensified and modular process control, showing that intensified RD designs impose narrower operational windows and stricter constraints. Multi-parametric MPC (mp-MPC) demonstrated superior disturbance rejection over P-I controllers, maintaining product purity. Modular RD in olefin metathesis improved operability via increased DOFs, allowing synchronized operations across parallel units but raising the total annual cost by 18.8 %. The study emphasized integrated design-control frameworks as key to optimizing dynamic performance, market adaptability, and sustainability in intensified processes.

Alcántara Avila et al. [82] conducted a comprehensive review on process intensification control, emphasizing the integration of optimization and control strategies to improve efficiency, sustainability, and dynamic operability. The study highlights the increasing complexity of intensified systems, necessitating advanced methodologies for real-time optimization. A key focus is on superstructure-based optimization, particularly MINLP, for systematically evaluating process configurations and optimizing RD systems. The review also discusses simultaneous design and control methodologies, such as MIDO, to enhance system resilience and disturbance management. Findings demonstrate that energy-efficient control strategies, including thermally coupled RD configurations, can achieve significant energy savings while maintaining robust performance. The study underscores the importance of integrating process intensification, optimization, and control to develop next-generation intensified systems with enhanced operational stability and economic feasibility.

Tian et al. [83] developed a simultaneous design and control framework for RD systems, integrating MIDO and Explicit MPC to enhance process intensification. The study introduced design-aware control, linking control laws to design variables such as column diameter and catalyst distribution. Applied to an MTBE RD system, this approach reduced total annualized cost by 7 % while maintaining 98 % bottom product purity under disturbances. By leveraging explicit MPC, the system identified 17 critical operating regions, ensuring rapid, computationally efficient responses to feed variations. The findings highlight how integrating control into the design phase enhances operability, economic performance, and robustness, representing a major step forward in process systems engineering.

Iftakher et al. [84] proposed an integrated design and control

framework for RD systems, using the driving force approach to optimize process performance and controllability. The study demonstrated that maximum driving force-based designs improved energy efficiency, reduced  $\rm CO_2$  emissions, and enhanced dynamic control. Through steady-state and dynamic simulations in Aspen Plus, multi-objective performance metrics were evaluated, including energy consumption, control indices (RGA, NI), and disturbance rejection capabilities. Results showed that optimized designs reduced energy consumption by 15 % and improved control efficiency by 20 %, maintaining minimal loop interaction (RGA  $\sim$ 1) and stable responses under P-I and MPC control. The study underscores the importance of integrating design and control methodologies to develop sustainable and operable intensified processes.

Iftakher et al. [85] introduced the RD-Toolbox, a computer-aided platform for integrating RD process design and control, addressing the complexities of intensified systems. This tool automates steady-state and dynamic simulations while enabling controllability evaluation. The study compared superstructure optimization and driving force-based methods for ETBE and ethyl acetate RD systems, showing that driving force-based designs reduced energy consumption by 25.2 % despite lower profit margins due to reduced production. Both designs demonstrated effective disturbance rejection under P-I and MPC, with RGA values near unity and NI confirming stability. In ethyl acetate production, the RD-Toolbox optimized a 15-stage column at maximum driving force, ensuring robust control performance. This tool represents a significant advancement in RD process development, systematically optimizing energy efficiency and control robustness.

Contreras-Zarazúa et al. [86] explored process intensification for biojet fuel production via the Alcohol-to-Jet (ATJ) pathway, replacing conventional oligomerization with a catalytic RD column. This intensified design achieved a 20 % reduction in TAC, a 50 % decrease in environmental impact (Eco-indicator 99), and a 22 % lower accident risk. The system directly produced hydrocarbons (C8-C16) meeting ASTM D7566–21 biojet fuel specifications, eliminating additional fractionation steps. Control studies validated the feasibility of P-I and MPC strategies, with P-I control demonstrating 30 % lower IAE and better disturbance rejection. While MPC exhibited advantages for complex scenarios, its performance was limited under large disturbances due to predictive model constraints. These findings confirm RD's potential to enhance efficiency, reduce environmental impact, and improve operational safety, making it a viable solution for sustainable aviation fuel production.

Moraru et al. [87] developed a plantwide control strategy for RD systems with recycle streams, focusing on material inventory balance, reaction stoichiometry maintenance, and ensuring production rate and product purity. The strategy was validated through dynamic simulations, where flowrate and composition variations were introduced to test the proposed control structure. The results demonstrated the system's ability to maintain operational stability under disturbances, ensuring process performance remains within the desired specifications.

#### 4.2.2. Extractive distillation (ED)

This section reviews recent studies on the control of ED systems, focusing on strategies for improving process stability, optimizing solvent selection, and enhancing energy efficiency. Advances in dynamic modeling and control methodologies are analyzed to address operational challenges and improve system performance.

Luyben [88] analyzed the impact of solvent selection on the dynamic controllability of ED processes, demonstrating that solvent properties significantly influence both steady-state economics and control performance. Using Aspen Plus and Aspen Dynamics, the study compared three solvents—water, dimethyl sulfoxide (DMSO), and chlorobenzene—for separating an acetone-methanol azeotropic system. DMSO exhibited superior control stability, achieving 99.95 % purity for both acetone and methanol, with a 7 % faster stabilization time (1 h vs. 1.5–2 h for other solvents). Control strategies involved dual temperature

control loops, with the chlorobenzene system requiring additional steam-to-feed ratio controllers due to its slow transient response. The findings emphasized that solvent selection should integrate dynamic controllability criteria, as solvents with optimal VLE properties enhance process stability and economic efficiency without introducing additional control complexities.

Wang et al. [89] optimized an ED system for methylal/methanol separation, evaluating control performance using Aspen Plus and Aspen Dynamics. The study compared two control structures: a fixed reflux ratio scheme and a reflux-to-feed (R/F) ratio strategy, demonstrating that the R/F control approach significantly improved disturbance rejection, effectively handling 20 % fluctuations in feed flow rate and composition. The optimized system, featuring an ED column with 52 stages and an entrainer recovery column with 22 stages, maintained a methylal purity of 99.9 wt % at an entrainer flow rate of 2900 kg/h. The R/F control strategy reduced total annualized cost (TAC) to \$615,390, highlighting its efficiency in enhancing both economic feasibility and dynamic stability in intensified separation processes.

Gil et al. [90] developed an ED process for ethanol dehydration using glycerol as an entrainer, integrating energy-efficient control strategies. The study identified optimal operating conditions, including a reflux ratio of 0.35 and an entrainer-to-feed molar ratio of 0.45, yielding significant energy savings. Two control strategies were implemented: entrainer makeup flow rate control for recovery column level regulation and entrainer feed flow rate control, with the latter demonstrating superior dynamic performance. Under feed composition and flow disturbances, the second strategy stabilized within 2–3 h, maintaining ethanol purity at  $\geq 99.5$  mol % with minimal temperature deviations ( $\leq 3$  °C). These findings highlight glycerol's viability as a sustainable entrainer, achieving operational excellence while reducing energy demand.

Luyben [91] designed an ED control strategy for  $CO_2$ /ethane separation in enhanced oil recovery (EOR) processes, addressing the challenge of a minimum-boiling azeotrope at cryogenic temperatures. A two-column configuration using natural gas liquid (NGL) solvent efficiently removed  $CO_2$  in the extractive column's distillate (95.57 mol % purity) while recovering  $C_2$  and heavier hydrocarbons in the second column. A plantwide control structure was developed, demonstrating that single-end temperature control was insufficient due to solvent-light key similarities. Instead, a composition controller regulating the reflux-to-feed ratio ensured product purity stability despite feed disturbances. Dynamic simulations confirmed stable reboiler and condenser duties (73.32 MW and 110.2 MW, respectively), proving that advanced control strategies can effectively manage azeotropic separations in industrial applications.

Ramírez-Márquez et al. [92] investigated dynamic control strategies for ethanol dehydration ED processes, comparing five distillation configurations, including conventional (CLR, CVR), side-stream (SSVR), and thermally coupled (DWC-TCLR, DWC-TCVR) systems. The SSVR configuration with glycerol demonstrated the best dynamic performance, achieving the lowest IAE values for ethanol and water composition control. Specifically, SSVR-GL exhibited reboiler duties of 4902.93 kW and an annualized capital cost of \$107.8k, outperforming traditional configurations in energy efficiency and controllability. Relative Gain Array (RGA) analysis revealed strong interactions across all systems, with DWC-TCLR and SSVR showing improved stability under RGA-based control loops. Additionally, glycerol exhibited lower toxicity, reduced CO2 emissions, and superior control behavior compared to ethylene glycol. These findings highlight the potential of side-stream and thermally coupled ED sequences in achieving sustainable and energy-efficient ethanol dehydration.

Ramos et al. [93] developed an optimal control strategy for ED in fuel-grade ethanol production, employing glycerol as an entrainer to enhance separation efficiency. The study applied dynamic optimization using a DAE model, discretized via orthogonal collocation, and solved through large-scale nonlinear programming in GAMS. Results demonstrated that optimizing reflux ratio and reboiler duty significantly

improved system stability, outperforming conventional P-I control under sinusoidal and step disturbances. The optimal control approach maintained product quality and economic profitability, achieving a 50 % net profit increase. Real-time feasibility was validated, with solution times as low as 9 s, highlighting the potential of dynamic optimization in process intensification.

Segovia-Hernández et al. [94] investigated the controllability of intensified bioethanol separation sequences, comparing conventional (CLR, CVR), side-stream (SSVR), and thermally coupled (TCLR, TCVR) ED systems. Using SVD, the study assessed system stability, showing that SSVR and CVR with glycerol exhibited higher minimum singular values and lower condition numbers, indicating superior dynamic robustness. Closed-loop P-I control simulations confirmed that SSVR-GL achieved the lowest IAE (0.00857) in ethanol purity control, outperforming thermally coupled sequences. These findings emphasize the role of solvent selection and process configuration in enhancing control performance and operability in intensified separation systems.

Errico et al. [95] introduced a two-column ED configuration for bioethanol purification, reducing capital costs by 10 % and energy consumption by 4.5 % compared to traditional three-column setups. Closed-loop dynamic analysis with P-I controllers showed superior stability, with the IAE for water control decreasing from 0.0929 to 0.000084. The optimized system achieved a 99 % ethanol recovery rate, outperforming conventional sequences while also reducing  $CO_2$  emissions (2.169 ton/h vs. 2.271 ton/h). These findings demonstrate that process intensification can enhance both economic and environmental performance, while maintaining robust control characteristics.

Luyben [96] compared the dynamic controllability of conventional and thermally coupled ternary ED systems, analyzing benzene, cyclohexane, and toluene separation. Aspen Dynamics simulations demonstrated that the thermally coupled system reduced reboiler duty by 14 % (3.632 MW vs. 4.230 MW) but exhibited inferior dynamic performance. The conventional system, using pressure-compensated temperature control, maintained 99 mol % benzene purity, while the thermally coupled design required composition control due to a flat temperature profile. Dynamic tests revealed greater purity deviations under feed disturbances in the thermally coupled system, highlighting the trade-off between economic efficiency and dynamic operability in intensified processes.

Ahmadian Behrooz [97] developed a robust control strategy for the ED of benzene-acetonitrile azeotropes, using dimethyl sulfoxide (DMSO) as a solvent. The study optimized fixed reflux ratio and fixed reflux-to-feed ratio control structures, demonstrating that reflux-to-feed ratio control improved regulatory performance, maintaining 99 % benzene and 99.9 % acetonitrile purity under Gaussian-distributed feed variations (mean 65 wt % benzene,  $\sigma=3.5$  wt %). Design modifications, including two additional plates and increased column diameter, provided a safety margin against flooding. The optimized fixed reflux-to-feed ratio structure (CS2) achieved faster transient responses, superior disturbance rejection, and only a 6.94 % increase in TAC, showcasing the effectiveness of integrating stochastic optimization with dynamic control in azeotropic separations.

Zheng et al. [98] analyzed the dynamic controllability of heat-integrated ED processes, comparing two novel configurations that integrate preconcentration and entrainer recovery. While achieving over 13 % energy savings, these systems introduced control challenges due to reduced degrees of freedom. Using SVD, the authors optimized temperature control tray selection and implemented three temperature control loops, which effectively managed  $\pm 20$  % feed flowrate and composition disturbances, maintaining product purity with reduced IAE values. The findings confirm that feed-forward ratio controllers enhance dynamic stability, making heat-integrated ED feasible for industrial applications.

Cao et al. [99] evaluated the economic and control performance of pressure-swing distillation (PSD) and ED for separating azeotropic systems in a varied-diameter column. The cascade control strategy (CS2)

for PSD, incorporating temperature and composition controllers, successfully maintained product purity (99.5 mol %) under  $\pm 20$  % disturbances, with settling times of 3 h. In contrast, ED required a more complex control configuration (CS4) and struggled with  $\pm 10$  % disturbances, stabilizing after 4 h. The study highlights that PSD with VDC outperforms ED in both control and economic feasibility, making it the preferred option for industrial applications with variable conditions.

Wang et al. [100] optimized the control structure for separating the ternary azeotropic mixture toluene-methanol-water via ED, comparing a three-column system and a two-column system with a decanter. The two-column process reduced TAC by 51.4 % while maintaining 99.9 mol % methanol purity. An improved control structure (CS4), integrating a proportional controller and an increased solvent flow rate, enhanced disturbance rejection. Increasing solvent flow to 85 kmol/h ensured stable operation, with only a 6.25 % increase in solvent use, demonstrating a balance between efficiency and controllability in process intensification.

Zhang et al. [101] designed and optimized control strategies for ED in ethyl acetate-ethanol separation, evaluating conventional and heat-integrated configurations. The B1-E configuration achieved 8.77 % energy savings and 4.38 % lower operating costs. Dynamic analysis identified single-end temperature control with a feed-forward strategy (CS3) as the most effective in reducing transient deviations. For heat-integrated systems, a bypass control scheme with dual-point temperature control ensured operational robustness under disturbances. These results emphasize that optimized control strategies are essential for enhancing the efficiency of intensified separation processes.

Luyben [102] examined the impact of pressure on solvent-to-feed (S/F) ratios and control performance in heat-integrated ED processes, comparing 1 atm and 10 atm configurations. The higher-pressure system reduced S/F ratio from 3.52 to 0.717, significantly cutting solvent flowrate (387 kmol/h vs. 1900 kmol/h) and reboiler duty (14.0 MW to 10.9 MW). However, the dynamic analysis revealed that the conventional reflux-to-distillate control strategy failed at 10 atm, causing oscillations and purity loss under feed composition variations. A modified reflux-to-feed control strategy improved stability, while an adaptive nonlinear solvent-to-feed controller successfully maintained methanol purity (99.5 mol %) under large disturbances. These findings underscore the importance of integrating advanced control strategies in high-pressure and heat-integrated ED processes to ensure stability and efficiency.

Jaime et al. [103] evaluated advanced control strategies for ethanol dehydration via ED, comparing a conventional scheme (Strategy 1) with a modified feedback-based control strategy (Strategy 2) regulating solvent flow and recovery column dynamics. Dynamic simulations confirmed that Strategy 2 achieved superior stabilization times (1–2 h vs. 3–5 h in Strategy 1), effectively managing glycerol concentrations to prevent hydraulic challenges linked to increased viscosity. Under feed disturbances (80–84 mol % ethanol), temperature variations remained within  $\leq$ 0.5 °C, and ethanol purity deviations were limited to 0.4 mol %, ensuring process stability and energy efficiency with a reboiler duty of 4281 kJ/s. These results underscore the effectiveness of dynamic control structures in managing non-linear, multivariable ED systems.

Das Neves et al. [104] developed an AI-driven control system for ED in anhydrous ethanol production, utilizing ANNs for real-time setpoint adjustments. The two-ANN model significantly improved control accuracy, reducing ISE from  $9.02\times10^{-8}$  to  $2.4\times10^{-8}$ , and achieved energy savings of 0.90 % and 0.94 % for -20 % and +20 % feed flow disturbances, respectively. Compared to conventional feedback controllers, ANN-based control minimized response times and enhanced disturbance rejection, proving its feasibility as an efficient and adaptive alternative for process intensification.

Pan et al. [105] integrated deep eutectic solvents (DES) and advanced control strategies for energy-efficient ethanol dehydration via ED. A multi-objective genetic algorithm optimized the system, reducing reboiler duty by 55 kW through waste heat recovery. Comparing five

control strategies, MPC outperformed P-I based schemes, reducing destroyed exergy by 58.43 kW. A ratio control strategy, derived from steady-state design simulations, enhanced feed composition disturbance rejection, enabling predictive adjustments and minimizing transient deviations. These findings highlight the role of model-based control in improving process stability and sustainability in intensified separations.

Ma et al. [106] analyzed the dynamic controllability of a side-stream ED process, focusing on response to feed disturbances. Conventional control structures failed under  $\pm 10$  % disturbances, necessitating an advanced cascade control strategy for side-stream composition and temperature regulation. While improving acetone purity stability, the method struggled with methanol purity deviations. A refined side-stream flowrate-to-feed flowrate ratio control achieved stable product purities within 16 h for  $\pm 10$  % disturbances, but remained ineffective for  $\pm 20$  % variations. The most effective strategy, integrating a composition controller and side-stream throughput valve, achieved stability within 8–12 h, handling  $\pm 20$  % feed rate fluctuations with delayed stabilization. Despite prolonged transients compared to conventional ED (1.5 h to steady state), the side-stream process improved energy efficiency, demonstrating the trade-offs between intensification, energy savings, and operational stability.

Ma et al. [107] reviewed dynamic control advancements in ED, emphasizing the integration of control strategies with process intensification techniques. The study explored Extractive Dividing-Wall Columns (EDWCs), achieving up to 11.6 % TAC reduction while enhancing energy efficiency. Advanced control strategies, including fuzzy-PID controllers, demonstrated improved robustness against feed disturbances, maintaining product purity with minimal deviations. The results highlight the importance of coupling design and control methodologies to optimize ED process stability and economic performance.

Yang et al. [108] optimized and controlled a Triple-Column Extractive Distillation (TCED) process for separating ethyl acetate, ethanol, and water, achieving a 14.11 % TAC reduction and 15.23 % lower exergy losses compared to conventional methods. Three control strategies were tested: fixed reflux ratio (CS1), dual temperature control (CS2), and feedforward-based control (CS3). CS3 exhibited superior disturbance rejection, maintaining 99.9 mol % product purity under  $\pm 10$  % feed variations, with stabilization times of  $\sim 3$  h. These findings highlight the effectiveness of integrated ED control strategies in enhancing operability and sustainability.

Zhang et al. [109] examined thermally coupled ED systems for separating THF, ethanol, and water, demonstrating higher energy efficiency and process stability. The integration of feedforward reboiler duty control and sensitive tray temperature adjustments minimized transient deviations and steady-state offsets. The intensified process achieved IAE values of 0.0012 under  $\pm 20$ % feed disturbances, significantly outperforming conventional systems. These results underscore the potential of thermally coupled ED for optimizing both economic and control performance.

Araújo Neto et al. [110] developed an intelligent control system (ICS) for ED-based ethanol production, leveraging ANNs to dynamically adjust setpoints. The soft sensor-based approach ensured seamless transitions to new steady states within 1-2 h, avoiding manual interventions and preventing overflow or emptying in reflux vessels. Simulation results showed that the ICS optimized solvent-to-feed ratios, reducing energy consumption while maintaining ethanol purity between 99.1 % and 99.9 %. These findings validate the feasibility of ANN-based control in industrial ED applications.

Zhang et al. [111] assessed dynamic control in vapor recompression-assisted ED, focusing on acetone-methanol separation. Traditional temperature control strategies exhibited significant product offsets under disturbances, prompting the development of a dual-impurity control strategy, regulating solvent-to-feed and reflux-to-distillate ratios. This optimized strategy reduced TAC by 20.53 % and energy consumption by 27.21 %, while vapor recompression cut energy use by 62 % and  $\rm CO_2$  emissions by 55.92 %. These results

highlight the necessity of plant-wide composition control for multivariable, heat-integrated ED processes.

Zhang et al. [112] proposed a double side-stream ternary ED configuration, achieving a 31.52 % TAC reduction while improving dynamic control performance. The temperature-cascade control loops effectively managed  $\pm 20$  % feed disturbances, restoring product compositions with minimal transient deviations. Partial heat integration and adaptive solvent flow regulation further enhanced operational flexibility, reducing TAC by 7.78 %–15.21 % in different case studies. Despite these advantages, challenges related to plumbing arrangements and control valve placements were noted, emphasizing the importance of integrating control robustness in process intensification strategies.

Neves et al. [113] developed an ANN-based intelligent control system for ED in anhydrous ethanol production, enhancing process stability and efficiency under simultaneous feed disturbances and product specification changes. The ANN-based controller dynamically adjusted setpoints in response to ethanol purity variations, significantly outperforming conventional P-I control. Steady-state errors were reduced up to 35 times, and setpoint adjustments were completed within 2–4 h. Trained with 1000 datasets, the ANN model captured process nonlinearities, ensuring robust disturbance rejection while maintaining minimum energy consumption, demonstrating the feasibility of AI-driven adaptive control in process intensification.

Liu et al. [114] optimized side-stream ED (SED) configurations for separating pressure-sensitive azeotropes, achieving a 6.6 % reduction in TAC and an 11.9 % decrease in CO<sub>2</sub> emissions compared to conventional designs. The SED1 configuration demonstrated superior operational safety, confirmed by the lowest Process Route Index (PRI). To enhance control performance, an MPC strategy was implemented, reducing IAE under dynamic disturbances, ensuring precise composition control, and outperforming P-I control in response time and robustness. These findings highlight the advantages of integrated design-control methodologies in energy-efficient intensified systems.

Wang et al. [115] developed an optimized control structure for a side-stream ED column used in methanol/toluene separation, achieving a 17.57 % TAC reduction and a 13.56 % decrease in energy consumption. Sensitivity analyses confirmed that optimizing entrainer concentration improved operational performance. Single Composition Control Structures (SCCS) outperformed Single Temperature Control Structures (STCS) in maintaining product purity under dynamic disturbances, demonstrating that composition-based control ensures greater process stability and economic efficiency in intensified ED systems.

Wu and Chien [116] proposed a cost-effective control strategy for hybrid reactive-extractive distillation (DCRED), eliminating composition analyzers by using temperature and temperature-difference (TD) controllers. Their optimized control structure (CS3) exhibited superior disturbance rejection, achieving IAE reductions from 63.6 to 8.45 for the TBA/EtOH/H<sub>2</sub>O system and from 1959 to 51.5 for the THF/EtOH/H<sub>2</sub>O system. The invariant TD control loops ensured stable product purity even under  $\pm 10\,$ % feed disturbances, demonstrating scalability and practicality for process intensification.

Torres Cantero et al. [117] evaluated four classical control structures (L, D, LV, and DV) for an ED column using  $CaCl_2$  as an entrainer in bioethanol production. Temperature-based inferential control strategies were tested via sensitivity analysis and SVD. Single-end structures (L and D) showed lower energy consumption (2.23 kW) and minimal ethanol purity deviations, while dual-end structures (LV and DV) exhibited faster transient responses but increased energy consumption, with DV requiring 200 % more reboiler duty. Error analysis confirmed L as the most energy-efficient structure, demonstrating the effectiveness of conventional control strategies in bioethanol dehydration.

Zhang et al. [118] integrated self-optimizing control into the design phase of ED processes, enhancing both economic efficiency and operational stability. Their optimized two-column configuration for acetonitrile-water separation achieved a 32 % TAC reduction, while a multi-objective optimization framework minimized temperature drift,

improving temperature stability by 38.7 % compared to conventional setups. By reducing the number of control loops, the study demonstrated simplified control structures with robust disturbance rejection, ensuring smooth process transitions and dynamic controllability, reinforcing the role of integrated design-control methodologies in PI.

Ge et al. [119] optimized ED for formic acid-water separation, achieving a  $27.01~\%~CO_2$  emissions reduction and a 23.45~%~TAC decrease compared to conventional methods. Their study demonstrated that MPC outperformed multi-loop P-I control, reducing overshoot, oscillations, and settling times by over 60 %. The optimized control strategy ensured 98 % formic acid purity and 99 % water purity, highlighting the critical role of advanced control frameworks in enhancing stability, efficiency, and sustainability in PI.

Neto et al. [120] investigated an intelligent control system based on ANNs for Indirect-Extractive Distillation (IED) in the separation of tetrahydrofuran, ethyl acetate, and water, a complex ternary azeotropic mixture. Compared to conventional control, the ANN-based system demonstrated superior performance in handling composition disturbances, significantly minimizing offsets from nominal product specifications. By automatically adjusting temperature profiles, the intelligent control system outperformed conventional approaches in terms of the IAE, ensuring enhanced dynamic stability with minimal human intervention. These findings highlight the scalability and robustness of ANN-based control for improving operational efficiency in intensified distillation processes.

#### 4.2.3. Dividing wall column (DWC)

This section provides an overview of research on the control of DWCs, highlighting developments in process integration, operational flexibility, and control structure design. The reviewed studies discuss approaches to improve product purity, energy consumption, and system robustness in intensified distillation processes.

Serra et al. [121] investigated advanced control strategies for DWCs, focusing on MIMO control structures to enhance process intensification. Their study optimized diagonal feedback and dynamic matrix control (DMCo) approaches for ternary separations, identifying the D-S-B paired control structure as the most robust, achieving a bandwidth frequency of 0.021 rad/min, a Morari resiliency index (MRI) of 0.65, and a condition number (CN) of 4.6. While DMC showed potential, it struggled with nonlinearity and slower convergence, requiring precise system identification. The research underscores the trade-off between energy optimization and controllability, highlighting that tailored control structures in DWCs are essential for operational stability and process efficiency in intensified distillation systems.

Adrian et al. [122] advanced DWC control by implementing MPC, demonstrating superior stability and disturbance rejection over conventional P-I controllers. For feed flow disturbances, MPC reduced temperature deviations from 6 to 8 K (PI) to 2–3 K and stabilization time from 12 to 2 h. Similarly, for feed composition disturbances, MPC limited deviations below 2 K and reduced stabilization time from over 10 h to 3 h. Experimental results from a miniplant-scale DWC confirmed that MPC effectively handled the strong multidimensional interactions inherent in DWCs, enabling operations closer to energy efficiency limits. Despite requiring three times the tuning effort compared to PI, MPC significantly improved dynamic performance and economic feasibility, reinforcing its potential for process intensification control.

Van Diggelen et al. [123] evaluated advanced multivariable control strategies for industrial DWCs, comparing PID controllers with LQG/LQR, GMC,  $H\infty$  control, and  $\mu$ -synthesis. While PID controllers maintained stability, they exhibited slow responses, with settling times exceeding 1000 min in some configurations. LQG with integral action reduced settling times to 510 min for feed disturbances, whereas  $\mu$ -synthesis proved the most robust, stabilizing product purities within 569 min while maintaining steady-state errors <0.002 in product composition. These results demonstrate that advanced multivariable controllers provide superior stability and efficiency, achieving up to 40

% energy savings and 30 % capital cost reductions, making them essential for sustainable process intensification.

Kiss and Rewagad [124] optimized energy-efficient control for benzene-toluene-xylene (BTX) separation in a DWC, evaluating PID-based multi-loop strategies. The DB/LSV control structure exhibited the best performance, with settling times under 7 h, compared to 14+h for LV/DSB. By introducing liquid split ratio (rL) optimization, they minimized energy requirements while maintaining 97 % product purity, achieving 40 % energy savings and 30 % lower capital costs. RGA analysis confirmed that DB/LSV had minimal process interactions, ensuring robust performance under  $\pm 10$  % disturbances. This study underscores that tailored control strategies are critical for maximizing efficiency and stability in DWCs, reinforcing their role in process intensification.

Kiss and Bildea [125] examined control challenges in DWCs, demonstrating that MPC outperforms multi-loop PID controllers for ternary separations such as benzene-toluene-xylene and pentane-hexane-heptane. MPC achieved shorter settling times and enhanced disturbance rejection, maintaining 97 % product purities while reducing energy consumption. In the benzene-toluene-xylene case, MPC minimized overshooting and handled 10 % feed composition variations more effectively than PID-based approaches. These findings emphasize the importance of advanced control strategies in overcoming the operational complexities of DWCs, ensuring sustainability and process efficiency in industrial process intensification.

Rewagad and Kiss [126] advanced dynamic optimization and control strategies for DWCs, emphasizing the superiority of MPC over conventional PID-based frameworks. Using benzene-toluene-xylene (BTX) separation, they demonstrated that MPC effectively handled feed flow (+10 %) and composition disturbances, maintaining 97 % product purities while optimizing energy use through liquid split manipulation. The IAE was consistently lower for MPC, highlighting faster disturbance rejection and stability. Additionally, hybrid MPC-PID control enhanced robustness and practical implementation, reinforcing MPC's potential for non-linear, high-degree-of-freedom systems like DWCs.

Tututi-Avila et al. [127] evaluated the dynamic controllability of an EDWC for ethanol dehydration, demonstrating a 13 % reduction in heating duties, 19 % lower cooling requirements, and 12.4 % TAC savings compared to conventional ED. Comparing fixed vs. adjustable vapor split control structures, they found that an adjustable vapor split significantly improved disturbance rejection and ethanol purity ( $\geq$ 99.5 wt. %). Dynamic simulations confirmed that adjustable vapor splits enhanced system stability and response times, validating EDWC as a viable intensified alternative for large-scale industrial applications.

Blevins et al. [128] implemented MPC in a pilot-scale DWC, demonstrating superior process control over PID controllers. MPC optimized temperature control, reducing variability to  $0.5^{\circ}$ F, and improved side-product purity from 0.8 to 0.9 mol fraction. WirelessHART transmitters and PIDPlus algorithms stabilized wireless control updates every 8 s, ensuring robust operation even under a 10 % feed reduction. These findings highlight MPC's transformative role in energy-efficient, high-performance DWC operations, setting a benchmark for process intensification.

Acosta-Solorzano et al. [129] analyzed bio-jet fuel and green diesel distillation sequences, comparing conventional (CDS, CIS) and thermally coupled (TCDS, TCIS, DWC) configurations. While TCDS achieved the lowest energy use ( $\sim$ 11 % savings), TCIS and DWC exhibited superior controllability, as indicated by lower IAE values. The TCIS structure balanced energy efficiency and dynamic stability, outperforming high-energy conventional designs. Control strategies such as PI tuning and relative gain array analysis were critical in ensuring operational stability while maximizing energy efficiency in biofuel separations.

Donahue et al. [130] provided an in-depth DWC control analysis, highlighting MPC as the most effective strategy over multi-loop PID and temperature-based control. Case studies from BASF (100+ DWCs) and

ExxonMobil showcased 30–50 % energy savings, but control challenges, particularly with liquid and vapor splits, could double energy consumption if misconfigured. Pilot-scale studies confirmed that MPC minimized offset and improved response times under feed composition variations, emphasizing the need for an integrated control framework to optimize DWC performance for industrial applications.

Qian et al. [131] analyzed stabilizing control structures for a three-product DWC, evaluating fixed liquid split (CS1), active liquid split (CS2), and active vapor split (CS3) strategies under  $\pm 20$  % disturbances in feed composition and flow rate. While CS1 demonstrated robustness in handling feed disturbances, CS2 and CS3 exhibited better disturbance rejection, particularly in stabilizing prefractionator temperatures. CS3 struggled with light component disturbances, whereas CS2 effectively managed variations through liquid split manipulation. The optimized DWC achieved 99 % purity in ethanol (distillate), n-propanol (side product), and n-butanol (bottom product), operating at 1 kmol/h feed flow and 30.86 kW reboiler duty. The study highlights that a DWC can be controlled using only three temperature controllers, eliminating the need for direct composition control, and offering a simpler and more cost-effective industrial implementation.

Tututi-Avila et al. [132] evaluated advanced control strategies for DWCs in BTX separation, comparing satellite, Kaibel, and conventional distillation sequences. Their study demonstrated that the satellite column achieved a 24.5 % energy reduction over the conventional sequence and an 11.8 % improvement over the Kaibel column, while maintaining stable dynamic responses. Composition controllers and liquid split manipulation were implemented, proving superior disturbance rejection for feed composition variations, with product purities recovering within six hours. The satellite column exhibited resilience to vapor split variations, stabilizing in five hours versus ten hours for the Kaibel column. These results validate DWCs as an energy-efficient and controllable alternative for petrochemical separations.

Sánchez-Ramírez et al. [133] performed a comprehensive control analysis of ten hybrid distillation designs for biobutanol separation, comparing conventional, thermally coupled, and intensified configurations. Using SVD and P-I controllers, they found that intensified designs exhibited superior control properties, with Design E achieving the best minimum singular value and lowest IAE for acetone control, while Design D excelled in butanol control. Intensified designs showed higher thermal coupling flow rates (118.62 kg/h liquid, 104.58 kg/h vapor in Design E), correlating with improved dynamic behavior and energy efficiency. These findings highlight the critical role of process intensification in achieving both lower energy consumption and enhanced control performance in biobutanol production.

Rodríguez et al. [134] explored control strategies for extractive and reactive DWCs, demonstrating that MPC outperformed decentralized control by reducing oscillations and effectively handling feed variations up to 5 %. Case studies included an extractive DWC for bioethanol dehydration and a reactive DWC for methyl acetate hydrolysis, where MPC stabilized ethanol and water compositions despite 2.5 % feed disturbances. The reactive DWC achieved stable methanol and acetic acid production with constrained dynamic adjustments (prediction horizon: 40 min, control horizon: 4 steps). These results confirm that MPC significantly enhances operational stability and efficiency in intensified separation processes.

Weinfeld et al. [135] provided a comprehensive review of RDWC advancements, emphasizing their potential for 25–40 % energy savings and 30 % lower capital costs. While major players like BASF operate over 70 DWCs, industrial adoption remains challenging due to complex control requirements and vapor-liquid equilibrium uncertainties. MPC and P-I control strategies have shown promising stability despite feed disturbances, and experimental validation confirms RDWC feasibility, with methyl acetate hydrolysis achieving 82.2 % conversion and ethyl acetate/methyl oleate separation reaching 92.1 wt % methanol purity. The study underscores RDWCs' transformative potential, bridging experimental progress and computational optimization toward

large-scale commercial viability.

Keil [136] provides a review of process intensification control, emphasizing innovations that enhance efficiency and sustainability. Heat-integrated distillation columns (HIDiC) reduce energy consumption by up to 50 %, while DWCs achieve 30 % energy savings by consolidating multi-step separations into a single unit. Microreactors improve heat and mass transfer rates, reducing reaction times by 90 %, and static mixers optimize continuous flow processes, lowering energy use by 40 %. The integration of multi-objective optimization frameworks in modular designs further enhances operational efficiency by balancing economic performance with environmental sustainability. This study underscores the critical role of intensified process control in optimizing chemical operations, reducing energy demand, and improving dynamic stability.

Wang et al. [137] developed an advanced sliding mode control (SMC) strategy for DWCs, optimizing process performance through response surface methodology (RSM) and particle swarm optimization (PSO). The study achieved 6.15 % energy savings compared to conventional single-factor optimization and demonstrated SMC's superiority over PID controllers, reducing settling time from 3.3 to 1.1 s for C5 composition tracking, with 12 times better performance under certain conditions. The SMC effectively managed nonlinear interactions and time delays, ensuring stable operation under feed disturbances and composition variations, positioning it as a robust control solution for highly integrated separation processes.

Lukač et al. [138] investigated the controllability of a four-product DWC with a 2–3–3 configuration, demonstrating its steady-state efficiency but highlighting dynamic challenges due to strong control loop interactions. Temperature-driven PID control struggled to maintain product specifications under feed disturbances, with IAE values reaching 13.2 and settling times ranging from 83 to 210 min. The study suggests that active vapor-split manipulation or advanced control schemes are necessary to improve dynamic stability and enable sustainable industrial adoption of this intensified design.

Zhang et al. [139] introduced advanced control strategies for liquid-only transfer Kaibel DWCs (LTS-KDWC) in a four-component alcohol separation process, comparing composition control (CS1), temperature control (CS2), and temperature difference control (CS3) under  $\pm 15$ % feed disturbances. CS3 outperformed other methods, maintaining 99 mol % purity across all streams while minimizing steady-state errors, demonstrating superior robustness and adaptability for industrial-scale implementation. These results underscore the importance of tailored control strategies in achieving high efficiency and operational stability in intensified separation processes.

Zhang et al. [140] proposed an LSTM-based MPC framework for an EDWC, addressing its nonlinear dynamics, multi-input multi-output nature, and time delays. The LSTM-MPC model was trained on extensive time-series data, ensuring high prediction accuracy with minimal mean squared error (MSE). Using multi-objective particle swarm optimization (MOPSO), the study determined optimal steady-state conditions, and three temperature inferential control (TIC) schemes were evaluated to identify the best input features for LSTM-MPC. Dynamic simulations tested the controller's performance under industrial disturbances, demonstrating superior closed-loop controllability, reduced offsets, negligible oscillations, and shorter transition times compared to conventional TIC strategies. These results highlight LSTM-MPC's potential for enhancing the stability and operational efficiency of EDWC, offering a more robust alternative for managing complex intensified separation processes.

Wang et al. [141] investigated the dynamic control of a liquid-only transfer extractive dividing-wall column (LTS-EDWC), optimizing its design and control performance for isopropanol-water-dimethyl sulf-oxide separation. The study first applies a multi-objective genetic algorithm to minimize total stage number and reboiler duty, followed by the development of three control structures. The basic control structure (CS1) stabilizes the system under  $\pm 10$  % disturbances but fails to reach

steady-state within 10 h. An improved control structure (CS2) achieves faster recovery under  $\pm 20$ % disturbances, maintaining product purity more effectively. To further enhance control performance, an adaptive neuro-fuzzy inference system (ANFIS)-PID controller (CS3) is introduced, reducing the time to steady-state by 8.6 % compared to CS2. However, CS2 proves to be more universally applicable across different extractive distillation systems, such as n-heptane-toluene-aniline and acetone-methanol-water, where it successfully restores product purity within 10 h under  $\pm 20$ % feed disturbances. These findings emphasize the potential of LTS-EDWC with optimized control strategies for achieving stable and efficient operation in intensified separation processes.

#### 4.2.4. Hybrid and membrane-based process control

This section examines recent works on the control of hybrid and membrane-based separation processes, addressing challenges related to process dynamics, mass transfer limitations, and system nonlinearity. The reviewed articles explore control strategies aimed at enhancing performance, stability, and energy efficiency in these integrated separation systems.

Iglesias et al. [142] presented a critical review of the integration of membrane technologies and photocatalysis for process intensification, focusing on advanced control strategies in photocatalytic membrane reactors (PMRs) and photocatalysis-membrane filtration (PMF) systems. These technologies enable simultaneous reaction and separation, enhancing energy efficiency and operational reliability in applications such as wastewater treatment and hydrogen production. Control innovations include UV irradiation optimization, tailored membrane coatings, and reactor geometry refinements to synergize reaction kinetics with membrane separation. Quantitative results demonstrate over 80 % total organic carbon (TOC) removal efficiency in wastewater applications, while PMRs for hydrogen production achieve H2 generation rates of up to 1000 µmol/g·h using Z-scheme configurations with Nafion membranes, surpassing conventional photocatalytic systems. These advancements address catalyst recovery, fouling, and mass transfer limitations, positioning PMRs as a key technology for process intensification, with ongoing challenges in scalability and long-term stability.

Jiang et al. [143] examined process control advancements in membrane crystallization (MCr), emphasizing precision in nucleation and growth regulation through novel membrane designs. The study highlights dynamic process control integration, optimizing interfacial mass transfer rates (0.66 mg/cm²/s) to achieve narrow crystal size distribution (CSD) and uniform morphology. Energy efficiency improvements of 20 %–30 % compared to conventional crystallization methods are demonstrated, with high packing density membranes (238 m²/m³) enhancing production efficiency and supersaturation control at nanometer precision. Case studies include pharmaceutical crystallization, where hollow fiber membranes regulate supersaturation, and hypersaline water treatment, achieving up to 84 % freshwater recovery and 2.72 kg/m²/day salt production. These findings underscore MCr's potential for sustainable crystallization, integrating advanced process control for optimized energy and material efficiency.

#### 4.2.5. Catalytic reactive systems

This section reviews key contributions in the control of catalytic reactive systems, focusing on strategies to optimize reaction performance, improve selectivity, and manage thermal effects. The discussion includes advanced control methodologies that address the complexities of reaction kinetics and multiphase interactions in catalytic processes.

Dautzenberg and Mukherjee [144] explored multifunctional reactors as an advanced PI strategy, integrating reaction and transport phenomena to enhance efficiency and sustainability. The study categorizes PI into four types: enhanced catalyst functionality (Type A), inter-phase transport intensification (Type B), intra-reactor process integration (Type C), and solid recirculation systems (Type D), demonstrating their role in improving heat and mass transfer, reducing energy consumption,

and minimizing by-products. Control strategies for these intensified systems remain a critical challenge, particularly in fluid catalytic cracking (FCC), where catalyst regeneration must be dynamically adjusted to prevent thermal instability, and catalytic distillation, where reaction and separation processes occur simultaneously. Quantitatively, FCC achieves over 75 % energy recovery, while catalytic distillation improves selectivity by 20–30 %. Recent innovations include microreactors, which leverage high heat transfer coefficients to enable ultra-fast reaction kinetics, and micro-engineered catalyst (MEC) systems, which optimize mass transfer while minimizing pressure drops. These findings underscore the necessity of adaptive control strategies to manage nonlinear dynamics and process interactions, ensuring operational stability and enhanced efficiency in PI applications.

De Toledo et al. [145] investigated process intensification control in bulk polymerization using an autorefrigerated continuous stirred-tank reactor (CSTR) with a semi-flooded horizontal condenser. The study addresses the challenge of non-condensable gas accumulation, which disrupts condenser pressure, reactor temperature, and heat exchange efficiency, leading to process instability. To counteract these issues, the authors propose an advanced control strategy incorporating periodic gas purging and compare P-I, OGPC-SQP, and adaptive STQGPC controllers. Quantitative results reveal that P-I control fails to stabilize the system, with oscillations exceeding  $\pm 5$  K, while QGPC and STQGPC reduce deviations to within  $\pm 0.5$  K, maintaining stable operation. Under a -10 % step perturbation in feed temperature, P-I control fails to restore stability, whereas predictive controllers recover steady-state conditions within 5000 s. These results highlight the necessity of advanced predictive control methodologies to optimize efficiency, ensure reliability, and enhance process safety in intensified polymerization systems.

Appel and Wachsen [146] developed an MPC framework for heterogeneously catalyzed gas-phase reactions, integrating a detailed kinetic model with statistical correlations from six years of industrial data. Their hybrid modeling approach, combining deterministic and statistical elements, optimizes reactor selectivity and stability, leading to 2 % selectivity improvements at the reactor exit under all operational conditions. The system incorporates real-time process optimization, automated data handling, and parallel simulations for scenario prediction, reducing material and energy costs while enhancing plant efficiency. Compared to traditional controllers, MPC enables more precise dosing adjustments and proactive decision-making, significantly improving reactor stability and operational robustness. These findings confirm that hybrid kinetic-statistical models, when integrated into MPC frameworks, provide scalable solutions for process intensification, particularly in selectivity optimization and long-term reactor stability.

Becht et al. [147] investigated microstructured catalytic wall reactors (MSRs) as a transformative process intensification strategy for highly exothermic gas-phase reactions, focusing on phthalic anhydride (PA) production from o-xylene. The study introduces a "booster concept," where MSRs handle 63 % of the total heat release within 20 % of the reactor volume, improving heat and mass transfer and process safety. Economic analysis indicates a potential 12 % reduction in production costs, with overall efficiencies 180 % higher than conventional reactors. Despite challenges in operational reliability and scale-up, the study establishes MSRs as a pioneering intensification strategy, offering significant energy savings, enhanced reaction control, and improved selectivity in chemical manufacturing.

Bahroun et al. [148] developed an advanced hierarchical control structure for high-pressure catalytic slurry reactors, specifically RAPTOR® for o-cresol hydrogenation. The system operates at 300 °C and 250 bar, requiring a Lyapunov-based controller with thermodynamic stability constraints to optimize conversions (98 %) while rejecting disturbances such as  $\pm 30$  % inlet gas flow variations. The control strategy stabilizes outlet temperatures and ensures operational safety, demonstrating robust disturbance rejection and energy efficiency. These results emphasize the importance of thermodynamic-based control in maintaining process stability and

optimizing efficiency in highly intensified reactors.

Ghiasy et al. [149] addressed pH control challenges in spinning disc reactors (SDRs), employing nonlinear compensation techniques such as pH characterizers and disturbance observers (DOs). While P-I control achieves an IAE of 10, it exhibits unacceptable overshoots (~40 %). Nonlinear compensation strategies eliminate limit cycles but at the cost of slower response times, whereas DOs enhance disturbance rejection. A case study on hydrochloric acid neutralization with sodium hydroxide highlights the fast dynamics of SDRs, where reduced size and enhanced mixing necessitate precise control strategies. These findings underscore the trade-off between responsiveness and stability in intensified chemical systems.

Li and Li [150] investigated neural network-based nonlinear MPC (NMPC) for three-phase catalytic slurry hydrogenation reactors, which operate under extreme conditions (300  $^{\circ}$ C, 250 bar). Two NMPC approaches are compared: SQP-based NMPC (NMPC-NO) and local linearization NMPC (NMPC-NPL). Results show that NMPC-NO achieves superior tracking accuracy (IAE = 9.14, ISE = 3.22) but higher computational costs, whereas NMPC-NPL reduces computation time by over 50 % while maintaining similar accuracy. Compared to linear MPC, NMPC exhibits better robustness under nonlinearities, highlighting its potential in optimizing control for highly intensified reactors.

Li and Li [151] extended their work by developing an MPC strategy for intensified continuous reactors, integrating a neural network-based Wiener model. Their hybrid approach simplifies NMPC into a computationally efficient linear MPC (LMPC), applied to hydrogenation of o-cresol at 200 bar and 300 °C. NMPC outperforms LMPC in setpoint tracking (IAE =5.84 vs. 6.63) under heat transfer variations, while LMPC slightly outperforms NMPC under mass transfer variations. These findings highlight the potential of NMPC in controlling intensified reactors, though online adaptation of neural network parameters is suggested to further improve robustness.

Kähm and Vassiliadis [152] introduced a stability criterion (K) for exothermic batch processes, embedding it into nonlinear MPC to prevent thermal runaways. By incorporating divergence-based stability constraints, their control scheme reduces reaction times by at least 50 % while ensuring stable operations at temperatures up to 450 K. Case studies show that stability-integrated MPC achieves optimized yields with reduced computational costs, cutting iteration times by up to 50 %. The approach is validated across multi-component reaction systems, where it predicts stability transitions with a 5 K margin before instability onset, enabling safe and efficient process intensification.

Jia et al. [153] explored process intensification control in cooling crystallization, integrating ultrasound, supersaturation control (SSC), and temperature cycling (TC) to optimize crystal morphology. Their combined approach achieves a 63.63 % increase in bulk density, L/D ratio reduction from 14 to 7, and uniform particle size distribution. Ultrasound enhances nucleation, SSC maintains steady growth, and TC eliminates fine crystals, collectively improving efficiency and sustainability in industrial crystallization. These findings highlight the critical role of process control in achieving intensified crystallization processes.

#### 4.2.6. Advanced control and optimization in process intensification

This section summarizes recent advances in control and optimization strategies for process intensification, with a focus on model predictive control, data-driven approaches, and real-time optimization techniques. The reviewed studies highlight methods to enhance operational efficiency, system responsiveness, and energy savings in intensified process systems.

Wang et al. [154] highlighted the quantitative benefits of PI in solid handling systems, including energy efficiency improvements of up to 30–40 %, process throughput enhancements of up to twofold, and operational cost reductions of approximately 20 %. These gains are achieved through enhanced mass and heat transfer rates, reduced process footprints, and shorter process times. The review emphasizes the critical role of advanced control strategies, such as MPC, in managing

the fast dynamics, sensitivity, and increased complexity of PI systems, with control performance metrics showing up to 50 % faster response times and improved process stability under dynamic conditions. These findings underscore the economic and operational advantages of PI when integrated with adaptive and real-time monitoring control systems.

Tian et al. [155] emphasized the quantitative integration of control systems within the design framework for process intensification. Utilizing multi-parametric model predictive control (mp-MPC), the study achieves optimal control strategies through dynamic optimization. Key outcomes include a closed-loop validation that maintains MTBE purity at 98 % under operational disturbances and uncertainties. Additionally, dynamic performance metrics such as Risk Ratios—0.91 for Operable Design 1 and 0.81 for Operable Design 2—highlight the role of control in reducing process risks. This approach ensures robust, economically optimized operations while enhancing flexibility and safety within intensified systems. The framework demonstrates the critical role of quantitative control analysis in achieving verifiable and operable designs in modern chemical engineering processes.

Lopez-Guajardo et al. [156] highlighted that recent advancements in Process Intensification 4.0 emphasize the integration of ML to enhance process control strategies, particularly through predictive and adaptive control systems. ML models, such as neural networks and hybrid physical-data approaches, enable real-time optimization of dynamic, non-linear processes, improving energy efficiency by up to 30 % and operational efficiency to exceed 95 %. The use of advanced sensors for real-time data collection further supports multi-parameter predictive control frameworks, facilitating robust performance under variable conditions. These innovations contribute to sustainable and circular processes by reducing waste and enhancing material recycling efficiency by 40 %, demonstrating the transformative potential of data-driven control in achieving resource-efficient operations.

Mokrova et al. [157] developed an approach for the intensification of automated control systems applied to industrial and transportation processes, achieving measurable improvements in efficiency and sustainability. In the production of activated carbon, they designed a control system for the drying and carbonization stages that utilizes recycled carbonization gases as a drying agent, resulting in a 25 % reduction in energy and operational costs. Additionally, they implemented an activation module with parallel-arranged furnaces and a centralized control system, optimizing performance and extending equipment lifespan by 20 %. In transportation systems, they applied predictive and dynamic models, including Mamdani systems, to manage urban traffic and reduce energy consumption in tunnel lighting by an average of 30 %. These advancements, based on hierarchical and decomposition principles, integrated hierarchical optimization methods and dynamic analysis to maximize operational and energy efficiency, demonstrating the applicability of their methodology across various industrial sectors with concrete results in energy savings and product quality enhancement.

These advancements are further supported by recent interdisciplinary developments that underscore the growing impact of AI and data-driven control across a wide range of complex industrial systems. For instance, the AI Institute in Dynamic Systems has proposed a common task framework for developing interpretable and physicsconstrained models through optimal sensor placement and the integration of data acquisition, modeling, and control. Wang et al. [158] introduced a differential geometry-reinforced learning framework (DGRL) that combines mechanistic and data-driven models, demonstrating superior control performance in highly nonlinear environments such as active suspension systems. Similarly, Mattera et al. [159] developed a reinforcement learning approach tailored to industrial manufacturing, including sim-to-real deployment in wire arc additive processes. Noriega and Pourrahimian [160] highlighted how AI and data analytics are transforming strategic mine planning through techniques like genetic algorithms and discrete simulations. Vake et al. [161] emphasized the role of open-source large language models in building

trustworthy and transparent AI systems with industrial applicability. Ghahramani et al. [162] proposed a smart manufacturing framework that leverages neural networks and evolutionary computing for intelligent automation in semiconductor production. In addition, Kutz et al. [163] contributed to the theoretical foundation of physics-informed machine learning by focusing on sparse regression, nonlinear dynamics identification, and interpretable model discovery. Although these studies are not limited to process intensification, they represent critical advances in AI-enabled control and offer transferable methodologies that can be adapted to the design and operation of intensified process systems.

To further ground these advancements, it is essential to move beyond performance metrics and examine the mechanistic foundations of control behavior in intensified systems. In particular, the nonlinear coupling between transport and reaction phenomena in RD and DWC units introduces stiff differential-algebraic equations (DAEs) that challenge traditional linear control assumptions. This has prompted the adoption of differential flatness and model order reduction (MOR) methods to derive tractable control-oriented models capable of preserving key physical dynamics while reducing computational burden [164].

In hybrid control architectures, mechanistic modeling plays a pivotal role when integrating soft sensors or AI modules. For example, observerbased estimation schemes grounded in reaction kinetics and mass transfer models can provide real-time inferential data to machine learning components, enhancing accuracy and stability. These hybrid estimators enable real-time reconciliation between empirical data and physical constraints, ensuring that AI models operate within feasible process envelopes and preserving thermodynamic consistency.

Furthermore, recent developments in Lyapunov-based stability analysis and passivity theory have been applied to validate control laws for intensified processes under uncertainty [165]. In NMPC frameworks, control Lyapunov functions are now routinely used to certify closed-loop stability, even in the presence of large input delays or non-minimum phase behaviors. These theoretical guarantees are particularly critical for self-optimizing control systems operating near safety-critical boundaries, such as in reactive crystallization or pressure-swing systems.

#### 4.3. Statistical analysis of control advancements (2000-2025)

A statistical evaluation was conducted to identify trends in process intensification control, revealing significant shifts in control strategies over the past 25 years. Fig. 1 illustrates the evolution of these techniques, highlighting the decline of traditional methods and the

increasing adoption of advanced control approaches.

One of the most notable trends is the decline in the usage of PID control. In the year 2000, PID controllers were employed in approximately 80 % of process intensification applications. However, their prevalence has significantly decreased over time, dropping to below 25 % by 2025. This decline can be attributed to the limitations of PID controllers in handling complex, nonlinear, and highly dynamic systems, which are characteristic of intensified processes. As a result, more advanced strategies, such as MPC and AI-based techniques, have gained prominence.

The adoption of MPC and NMPC has shown a steady rise, increasing from just 20 % in 2000 to approximately 45 % in 2025. These techniques have been particularly beneficial in applications such as RD, ED, and DWCs, where dynamic optimization and predictive capabilities provide substantial performance improvements. The ability of MPC and NMPC to handle multivariable interactions and constraints makes them particularly well-suited for complex intensified processes.

AI-based control strategies, including ANN and soft sensors, have emerged as a viable alternative in recent years. These techniques began gaining traction around 2015 and have since grown to account for approximately 20 % of the control strategies employed in 2025. AI-based control methods offer enhanced real-time adaptability, robustness against uncertainties, and improved predictive capabilities, which are essential for modern process intensification applications. By leveraging machine learning algorithms, these methods can optimize control actions dynamically, ensuring more stable and efficient process operations.

Another key trend is the rise of hybrid control strategies, which integrate model-based approaches with AI techniques. The adoption of hybrid control has expanded significantly, from just 5 % in 2010 to 25 % in 2025. This approach combines the advantages of traditional model-driven strategies, such as MPC, with the learning capabilities and adaptability of AI-based systems. Hybrid control frameworks enable better handling of nonlinearities, disturbances, and uncertainties, making them an attractive solution for next-generation process intensification.

Overall, these trends indicate a transformative shift in process control methodologies, moving away from conventional PID controllers towards more intelligent, adaptive, and predictive approaches. The increasing reliance on MPC, NMPC, AI-based control, and hybrid strategies reflects the growing complexity of process intensification and the need for more sophisticated control solutions to ensure efficiency, stability, and sustainability in industrial applications.

Fig. 2 provides a multidimensional representation of the evolution of

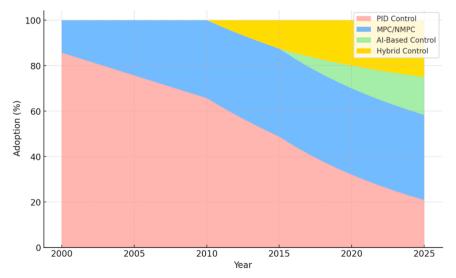


Fig. 1. Evolution of control strategies in process intensification: transition from traditional to advanced approaches.

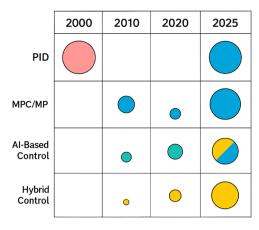


Fig. 2. Visual mapping of technological shifts in control strategies for process intensification (2000–2025).

control strategies in process intensification between 2000 and 2025. The matrix captures the relative intensity and timing of adoption across four major control paradigms: PID, MPC/NMPC, AI-based, and hybrid systems. The size of each colored circle is proportional to the relative prevalence reported in the literature for each time interval, allowing for a visual comparison of both temporal trends and the co-existence of multiple strategies. This format offers a complementary perspective to statistical plots by explicitly illustrating the overlapping implementation of different techniques during transitional periods in the field.

Color coding is used to distinguish the primary control approaches: red for PID controllers, blue for MPC and NMPC techniques, green for AI-based strategies including neural networks and reinforcement learning, and yellow for hybrid control frameworks that combine model-based and data-driven methodologies. The diagram emphasizes the diminishing dominance of PID as more advanced methods gained traction, particularly in systems with strong nonlinearities and real-time optimization requirements. The visual clustering of AI and hybrid methods after 2015 reflects the growing accessibility of computational resources and the integration of machine learning into industrial automation. Together with the quantitative data of Fig. 1, this figure reinforces the observed shift toward intelligent, predictive, and sustainable control architectures in intensified process systems.

#### 4.4. Impact on efficiency and sustainability

The impact of advanced control strategies on efficiency and sustainability in process intensification has been widely studied, revealing significant improvements in energy consumption, economic performance, and operational stability. Across multiple reviewed studies, the implementation of intensified process control strategies has led to substantial energy savings, with reductions ranging from 15 % to 40 %. These savings are primarily achieved through optimized process dynamics, enhanced heat integration, and improved system responsiveness, which collectively contribute to lower energy consumption and reduced carbon footprints in industrial applications.

From an economic perspective, the adoption of modern control techniques has translated into notable reductions in TAC. Depending on the control strategy employed and the specific process type, cost reductions have been reported in the range of 12 % to 30 %. MPC, NMPC, and AI-driven approaches contribute to these savings by optimizing resource utilization, minimizing waste, and enhancing process efficiency. The ability to dynamically adjust operational parameters in real time allows these strategies to maintain optimal performance, thereby reducing operating costs and improving overall profitability.

In addition to economic and energy benefits, intensified control strategies have significantly improved operational stability. Advanced control methods, particularly NMPC and AI-driven controllers, have demonstrated superior disturbance rejection capabilities, reducing the impact of process variations and external disruptions. These approaches have also been shown to decrease stabilization times, enabling processes to recover more rapidly from perturbations and maintain steady-state operation more effectively. The integration of predictive and adaptive control techniques ensures a more robust and resilient process, ultimately leading to higher efficiency and sustainability in industrial applications.

#### 5. Sustainability and process intensification control

The integration of sustainability into PI requires a fundamental shift in how control systems are conceived and implemented. Traditional methods that prioritize operational efficiency and product yield must evolve to address the broader environmental and social impacts of industrial processes. By embedding sustainability metrics into control frameworks, PI can transcend conventional optimization paradigms and become a cornerstone of green manufacturing.

A key principle of green engineering is the integration of process monitoring and control to enhance efficiency and sustainability in chemical manufacturing. As highlighted by Jiménez-González & Constable [166], effective control strategies, such as real-time optimization and advanced regulatory control, contribute to minimizing waste, reducing energy consumption, and improving resource utilization. By ensuring stable operation within optimal conditions, control systems prevent deviations that could lead to increased emissions or inefficiencies. This aligns with the broader objectives of green chemistry and engineering, demonstrating that well-designed control strategies are not merely operational necessities but fundamental components in the transition toward more sustainable industrial practices.

Achieving this transformation starts with real-time monitoring systems capable of assessing the environmental footprint of every operational decision. Advances in digital twins and life cycle assessment (LCA) algorithms have enabled process engineers to evaluate metrics like greenhouse gas emissions, water usage, and material efficiency dynamically. For instance, incorporating LCA feedback loops into MPC systems allows for decision-making that minimizes environmental impacts without compromising performance.

A key enabler of sustainable PI is the use of adaptive control strategies to accommodate renewable energy sources. The variability inherent in solar and wind energy often disrupts the stability of traditional processes. However, AI-driven controllers equipped with reinforcement learning capabilities can adapt process conditions in real time, ensuring seamless integration of renewable energy inputs. Such systems have been shown to enhance the efficiency of energy-intensive operations, including separations and catalytic reactions, by dynamically optimizing energy use based on availability.

Decentralized control architectures represent another leap forward for sustainability in PI. These systems distribute computational and decision-making power across modular units, reducing vulnerabilities associated with centralized systems. Decentralization also facilitates localized optimization, where individual modules adjust their operations to achieve global sustainability goals. Recent advancements in self-healing control systems, leveraging distributed ledger technologies and AI, further enhance resilience by enabling processes to detect and recover from faults autonomously.

The future of PI control may also hinge on the application of quantum computing. With its unparalleled computational power, quantum algorithms offer the potential to solve complex optimization problems that are currently beyond the reach of classical computing. This capability is particularly relevant for PI systems, where the interplay of multiple variables in nonlinear processes demands high-speed, high-accuracy solutions. Quantum-enabled control could revolutionize areas such as energy recovery, waste minimization, and material efficiency.

To fully realize the potential of sustainability in PI, interdisciplinary collaboration is essential. Material scientists, data engineers, and

process designers must work together to create systems that are not only efficient but also aligned with global sustainability goals. By integrating circular economy principles, leveraging AI and quantum computing, and embracing decentralized architectures, PI can pave the way for a future where industrial processes are both economically and environmentally sustainable.

An emerging direction in sustainable PI control involves integrating supervisory control layers specifically designed to monitor environmental performance metrics. These higher-level systems operate over standard real-time controllers, continuously tracking indicators such as resource circularity, carbon intensity, and cumulative energy demand. By enabling the system to anticipate and correct deviations from sustainability targets, this hierarchical architecture bridges the gap between process optimization and long-term ecological objectives.

#### 6. Future perspectives

The control of intensified processes is undergoing a paradigm shift, driven by emerging technologies that not only integrate seamlessly into existing systems but also redefine the design, management, and optimization of industrial operations. Several innovative areas have demonstrated the potential to address current challenges while advancing efficiency, adaptability, and sustainability.

Generative AI is reshaping the development of control strategies by enabling the creation of operational models capable of predicting system behavior and proposing real-time optimal configurations. Combined with digital twin technologies, these tools provide precise virtual environments to simulate and validate control strategies before physical deployment. Their application has proven particularly effective in complex industrial settings, including advanced chemical production and energy-intensive operations.

Meta-learning in process control has emerged as a transformative approach. This methodology enables algorithms to adapt swiftly to new data and operational scenarios with minimal external intervention. When applied to predictive control systems, meta-learning enhances automation in highly dynamic and nonlinear processes, such as plasma reactors and thermal-gradient-based separation systems, significantly improving adaptability and robustness.

The integration of quantum-based sensors represents a major advancement in process monitoring accuracy. These sensors offer unprecedented sensitivity, enabling the detection of microfluctuations in real time—critical for maintaining stability in intensified processes. This innovation is particularly beneficial for systems operating under extreme conditions, such as those involved in high-pressure and high-temperature synthetic fuel production using green hydrogen.

In terms of sustainability, advancements in multi-objective optimization models facilitate the effective alignment of economic, environmental, and operational goals. These models incorporate metrics such as carbon footprint, water usage, and energy efficiency, providing integrated decision-making tools that meet the increasing demand for sustainable industrial practices.

The development of autonomous systems guided by algorithmic ethics is gaining momentum in industrial process design. These systems consider operational efficiency alongside parameters related to safety and social impact. For instance, advanced chemical plants are deploying algorithms designed to mitigate risks associated with human exposure while ensuring safe and equitable working conditions.

These innovations collectively signify a transformative shift in the approach to controlling intensified processes. By addressing present challenges and integrating forward-thinking methodologies, the field is advancing toward a future defined by enhanced efficiency, resilience, and sustainability.

To ensure that these emerging control strategies are broadly adopted and rigorously validated, the availability of open-access datasets and standardized benchmark problems is becoming increasingly important. These resources allow researchers and practitioners to test hybrid and AI-driven control systems under comparable conditions, facilitating reproducibility and accelerating innovation. For instance, efforts like the AI Institute for Dynamic Systems have begun developing structured datasets and task frameworks specifically for physically-constrained control applications, serving as a foundation for benchmarking across sectors.

Such datasets are particularly critical for hybrid control systems, where integrating first-principles models with machine learning demands systematic evaluation of accuracy, generalizability, and computational performance. Including benchmark scenarios for common intensified operations (such as dividing-wall distillation, membrane-reactive systems, or heat-integrated reactors) would enable the community to assess the scalability and robustness of proposed solutions in realistic settings. In turn, this would support the industrial deployment of adaptive and autonomous control schemes with greater confidence.

#### 7. Conclusion

The evolution of control strategies in process intensification reflects a broader transformation in industrial process engineering, where adaptability, predictive capabilities, and sustainability have become key priorities. Traditional control methods, such as PID controllers, once dominant, are increasingly inadequate for managing the complexity of modern intensified processes. The growing reliance on advanced techniques like MPC, Nonlinear MPC, and AI-driven approaches signifies a shift towards more intelligent, self-optimizing control architectures capable of handling multi-variable interactions, nonlinear behaviors, and real-time decision-making.

One of the most significant impacts of this transition is the enhanced integration of control systems with process design. Unlike conventional approaches, which often treat control as an afterthought, modern strategies emphasize a co-optimization framework where control performance is considered during the design phase. This shift leads to inherently more stable, efficient, and flexible process configurations, reducing the need for costly retrofits and enabling a more seamless adaptation to fluctuating operating conditions. Hybrid control systems, which blend model-based and AI-driven techniques, exemplify this trend by ensuring robust process behavior even under uncertainty, demonstrating the growing role of machine learning in industrial automation.

Beyond efficiency and cost savings, these advancements have profound implications for sustainability. As industries strive to meet stricter environmental regulations and carbon reduction targets, optimized control strategies play a crucial role in minimizing waste, reducing emissions, and improving energy efficiency. The ability to dynamically adjust process parameters in response to real-time data not only enhances performance but also enables more sustainable use of resources. This is particularly critical in energy-intensive processes such as RD and DWC, where advanced control methods have demonstrated significant reductions in energy consumption and environmental impact.

The diversification of control strategies across different intensified processes highlights the need for tailored solutions rather than a one-size-fits-all approach. Processes such as ED and Hybrid Membrane-Reactors benefit from targeted methodologies that align with their specific operational challenges. The increasing adoption of AI-based control in Catalytic Reactive Systems suggests that future innovations will likely focus on autonomous and self-learning systems, capable of continuously improving performance without human intervention.

However, several current bottlenecks still limit the broader deployment of advanced control in process intensification. These include model uncertainty, sensor limitations, lack of interoperability with legacy systems, and computational burdens associated with real-time optimization in large-scale nonlinear systems. Furthermore, the gap between control design and sustainability metrics remains a critical area of concern.

Looking ahead, the integration of digital twins, reinforcement

learning, and real-time optimization into process control frameworks presents new opportunities for further enhancing efficiency and resilience. The challenge will be ensuring that these technologies can be implemented in a scalable, cost-effective manner while maintaining operational reliability. Additionally, as regulatory frameworks evolve, industries will need to balance innovation with compliance, ensuring that advanced control methodologies align with safety and environmental standards.

In this context, future research should also explore computationally efficient architectures capable of scaling with the complexity of large PI systems. Distributed and decentralized control strategies offer promising pathways to reduce centralized computational burdens while enhancing fault tolerance and modularity. Similarly, edge-AI frameworks can enable localized, low-latency decision-making, reducing dependence on cloud infrastructures and facilitating real-time control in network-constrained environments. These approaches hold particular potential for modular PI units and geographically distributed chemical production schemes.

In light of these limitations, future research should focus on four critical areas: (i) developing interpretable and certifiable AI models that align with process safety requirements, (ii) embedding lifecycle-based sustainability indicators into control objectives, (iii) enabling adaptive control in systems powered by intermittent renewable energy sources, and (iv) establishing robust co-simulation environments that bridge design, control, and economic decision-making in intensified operations.

In conclusion, the trajectory of control strategies in process intensification underscores a fundamental shift towards smarter, more sustainable industrial operations. The continued convergence of process design, control, and AI-driven optimization will be pivotal in shaping the next generation of intensified chemical processes, positioning them at the forefront of efficiency, adaptability, and environmental responsibility.

#### CRediT authorship contribution statement

César Ramírez-Márquez: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. José María Ponce-Ortega: Writing – original draft, Supervision, Project administration, Methodology, Investigation, Formal analysis, Conceptualization. Juan Gabriel Segovia-Hernández: Writing – original draft, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Salvador Hernández: Writing – original draft, Visualization, Validation, Supervision, Methodology, Investigation, Data curation, Conceptualization.

#### 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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#### Data availability

Data will be made available on request.

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