Process Intensification – An Insight

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Genesis

The process economies of chemical industry are fraught with two major challenges namely, capital investment and operational expenses. While one could be reduced at the cost of the other, simultaneous reduction in both the costs would result in an optimal solution. Improving process efficiency to lower the equipment volumes and simultaneously combining a number of unit operations in an equipment would bring down associated capital investment. Process Intensification typically addresses these aspects of cost optimization either by miniaturization of equipment or by synergizing various process steps. Process intensification (PI) can be defined as a set of innovative technologies that focuses on minimization of plant profile thus reducing capital costs and their integration to achieve high capacity, reduced footprint (area), increased selectivity and enhanced heat and mass transfer rates.¹ It involves constant miniaturization and integration of engineering methods and equipment.⁷ Its approach towards design of sustainable chemical processing plants is based on four dimensions namely, structure, energy, synergy and time.²

The term, 'Process Intensification' was introduced in 1960s by an East-European publication on Metallurgy. In 1983, Prof. Colin Ramshaw, widely regarded as the father of process intensification (PI), developed first set of innovations related to the subject in separation processes.^{1,2} His work analyzed the effect of high gravity field in distillation and a hybrid strategy to combine separation with reaction processes in a single column packed with catalyst.¹ In the 10th Annual Research Meeting of Institute of Chemical Engineers (IChemE) on process intensification held in the same year, the first research paper from South Africa was based on intensive underground processing of gold ore.³ A total of 16 papers concerning the fields of mineral processing, heat transfer, chemical reactor and plant design with control were presented in the aforesaid meeting.³ On a commercial scale, Eastman Chemicals set up a reactive distillation tower in 1980s with multifunctional capabilities reducing 80% energy requirements as compared to conventional operations.⁴ Throughout the next two decades, mostly within United Kingdom, a lot of improvised versions of PI sprung up utilizing the concepts of centrifugation, enhanced heat transfer based on spatial domain and micromixing. In 1995, the first full conference on PI for chemical industry was organized in Antwerp, Belgium by UK-

based BHR Group, a technology organization in fluids engineering.² Towards late nineties PI technologies were developed in research labs across the world such as microreactors in USA (Massachusetts Institute of Technology) and Germany (Institut für Mikrotechnik, Mainz), high-gravity processing in China (Beijing University), compact heat transfer in France (Greeh CEN Institute) and centrifugal adsorption in The Netherlands (TU Delft).⁵ The concepts of PI in chemical industry has started evolving quite strongly in the past 15 years with interestingly novel hardware and software applications being reported in bio-processing and fine chemicals.^{5,6}

2.0. Why Process Intensification?

The cardinal principle governing process intensification has been improved utilization of molecular interactions under optimized conditions of driving forces and synergistic effect of different unit operations. While gradient-free temperature and concentration profiles are achieved with reduction in residence time,² lower pressure level can be maintained with minimum energy requirements thus ensuring high level of safety in chemical plants. The combination of different unit operations results in better process control with lower degrees of freedom.² Adopting PI techniques results in nearly 20% of total plant cost.¹ Operating costs largely comprise of the consumables, process utilities and level of plant maintenance. These parameters can be optimized with improved output yield and turnaround time achieved through PI techniques. The opportunities offered by PI are summarized as shown in Fig. 1.0.

Fig. 1.0: Main advantages of process intensification

Table 1.0 summarizes various alternative sources and forms of energy that can be utilized for PI technologies in order to attain gross efficiency enhancement of chemical and biochemical processes.⁸ PI gains momentum in commercial operations for reduced requirement for consumables as well as waste management. The benefits of reduction in plant economics and energy requirements as achieved in the petrochemical industry have been summarized in Table 2.0 by the researcher from Shell Technology Solutions, The Netherlands.⁹ As per an European publication¹⁰ in 2008, the overall energy savings across industrial sectors (namely bulk & fine chemicals and food processing) on use of PI technologies such as multifunctional equipment, micro-reactors, microwaves and high gravity field (spinning tube reactor) is estimated (on current trends) to reach nearly 100×10^{15} Joules per year by 2050.

Table 1.0: Process intensification effects of the alternative sources and forms of energy¹⁰

Source of energy	Intensified element	Intensification factor [*]
High gravity field	Reaction time	1000
Static electric field	Interfacial area (by emulsification)	500
Microwaves	Reaction time (for liquid-phase catalytic reaction)	1250
Ultrasound irradiation	Reaction time	25
Supersonic flow	Gas-liquid mass transfer coefficient	10
	Capacity (for fluidized-bed reactor)	2.25

^{*} Reported possible improvement (as number of times) in comparison to conventional technology

Table 2.0: Process intensification in petrochemical industry and their benefits⁹

Technologies	Benefits			Commercial implementation [*]
	Feedstock cost	Capital cost	Energy requirements	
Reactive distillation		20-80%	20-80%	>150
DWC distillation		10-30%	10-30%	>100
Reverse flow reactor		>20%	Low	>100
Microchannel reactor	Yes	Yes (for small scale)		Only in fine chemicals and pharmaceutical sector

^{*} In terms of number of units commercialized [6][9]

3.0 Principles of Process Intensification

The major principles that drive the process intensification are as follows:

3.1 Maximizing effectiveness of intra & intermolecular events

- The processes not limited by their inherent kinetics but governed by changing the kinetics
- Improving conversions & selectivity
- Avoiding unwanted side products
- Improving number & frequency of collisions
- Better understanding of equipment & flow geometry for improved orientation of molecules in the moment of collisions & their energy
- Designing uniform products with minimum waste
- Macroscopic residence time distribution, reduction in dead zones & bypassing
- Meso & micro-mixing

3.2 Optimizing driving forces & maximizing specific area

- Improving transport rates across interfaces by maximizing interfacial area from millimeter to the micrometer scale of channel dimensions
- Maximizing driving force (concentration difference) not always required

3.3 Maximizing synergistic effects from partial processes

- Multifunctionality on the macroscale
- Reaction-cum-separation by removing products from the reaction environment

Process intensification differs from process optimization (defined as performance improvement of existing concepts) and process system engineering (defined as multi-scale integration of existing and new concepts). Table 3.0 gives brief description of each process.

Table 3.0: Basic Features of Three Areas of Chemical and Process Engineering

	Process Optimization	Process Systems Engineering	Process Intensification
Aim	Performance improvement of existing concepts	Multi-scale integration of existing and new concepts	Development of new concepts of process steps and equipment
Focus	Model, numerical method	Model, software	Experiment, phenomenon, inter-phase
Interdisciplinarity	Weak (interface with applied mathematics)	Modest (mostly applied mathematics and informatics, chemistry)	Strong (chemistry & catalysis, applied physics, mechanical engineering, materials science, electronics etc.)

4.0 Barriers to Process Intensification

While several PI technologies have achieved success on a lab scale, they are still at infancy as long as their industrial applications are concerned. The industrial adoption of PI is faced with several barriers:

- Lack of in depth knowledge and know-how in PI among process engineers
- Inadequacy in pilot scale facilities for PI for the existing production lines
- High stakes (both technical and financial) in prototype development as well as in retrofitting of existing process
- Lack of awareness among the top management about the potential benefits of PI technologies
- Non-availability of process control systems catering to novel PI modules

The R&D efforts in chemical industry primarily focus on new products (chemistry) and much less on the new manufacturing methods (chemical engineering). The chemical manufacturers are not interested in developing novel types of equipment or processing techniques and simultaneously the equipment manufacturers and engineering companies are not sufficiently active in process intensification.

Other barriers include high cost to retrofit PI technologies in current plants thus increasing the risks of commercializing breakthrough technology. Scaling up of PI techniques from lab to pilot to commercial scales poses major challenges. PI technologies warrant long development path, which calls for effective IP cooperation among various R&D labs, universities and industries.

5.0 Process Intensification – Select Technologies

The technology application for PI broadly include *micro-reactors, simulated moving bed reactor, oscillatory baffled reactor, tube-in-tube reactor, reactive distillation, membrane or ultra-sound crystallization and compact heat exchangers*. PI has been successfully applied for scale-up of continuous reactors and also for alternative retrofit methods for existing processing plants. Fig. 2.0 presents common classification for PI technologies as suggested by Stankiewicz and Moulijn.⁷ Numerous case studies elaborate the advances in PI over the past three decades that started off with the reactive distillation process developed in 1983.^{1,2} The merits of reactive distillation include reduced equipment size thus lowering capital cost, less energy use, and higher product yields.⁴ Recently, M. Errico et al. suggested a retrofit to existing distillation systems through thermal coupling technique to minimize physical size of the plant. It involved elimination of a few number of condensers and reboilers involving the sub-mixtures that resulted in a reduction of 23% in energy costs when compared to the existing plant design layout.¹¹

Fig. 2.0: Broad technology applications for process intensification.⁷

5.1 Micro-reactors

Micro-reactors is a class of chemical reactors at sub-millimeter level that can achieve reduced volume, higher product selectivity and higher reaction rate.^{2,5} It helps in complete mixing even for highly viscous fluids at large flow rates. Alternative reaction pathways are made possible due to higher heat and mass transfer rates and precise control of reaction volume under consideration.^{5,12} Figs. 3.0 (a) and (b) are schematic representations of pioneering attempt on an integration of automated process controls and micro-reactors for homogeneous catalysis.¹³ Typical applications include catalysis, steam reforming, organic synthesis and multiphase reactions.^{13,14} A recent example has been the use of cross-flow micro-channel reactors for reversible polymerization at 90°C to form poly(vinyl butyral), a high-demand product for the manufacture of safety glasses in automobile industry.¹³ It involves viscous fluids such as poly(vinyl alcohol) and n-butanal in presence of acid catalyst. Conventionally the batch process has lower rate of reaction with a product yield of 20 to 30% (expressed in terms of butyral group content) resulting in precipitation problems and phase change. On using 1000 µm length x 500 µm wide micro-channels made of stainless steel and PTFE (Teflon) in cross-flow pattern submerged in water, a well-controlled reaction takes place at 50°C within less than 56 seconds and the product yield has been reported as high as nearly 75% after an hour of aging.¹³ The optimized flow-rate at 5% PVA solution for a maximum limit of 80 ml/min resulted in good mixing performance as shown in Fig. 4.0 (c).¹³ The resultant process optimization for catalyst can lead to reduction of working capital for different utilities. Some of its major limitations

include insignificance of gravity forces, scale formation due to material incompatibility and micro-fabrication costs.

Fig. 3.0: (a) Schematic view of microreactors as printed circuit boards¹⁴ (b) ‘Numbered-up’ approach of multiple microreactors¹⁴ (c) Comparison of product yield (% in terms of acetalization degree (AD) or butyral group content) for the reversible polymerization reaction at a mass concentration ratio of n-butanal to PVA of 0.8 and an aging time (ω) of 3 hours¹³

5.2 Membrane Distillation

Role of membrane technology in enhancing mass transfer efficiencies has been significant by reduction in capacity and energy requirements. It is a hybrid technique combining membrane separation and conventional distillation.⁷ Desalination units based on osmotic pressure difference cover nearly 60% of existing plants all over world.¹⁵ But their performance suffers due to brackish feed water with high salt content. Recently, direct contact membrane distillation with conductive heat transfer is reported to have achieved nearly 100% inorganic salt rejection with no additional energy consumption.¹⁶ The technique developed by Hickenbottom and Cath for feeds with very high total dissolved solids (TDS) utilizes temperature and flow reversals on alternate basis to control variations in water flux and salt concentration profile. It uses a thin Teflon (TS22) active stratum and polypropylene (PP22) support layer of overall thickness 175 μm , effective surface area 89 cm^2 , pore size 0.22 μm and porosity of 70%. The pre-filtered water is fed at 1.6 Lits/min at 50°C and the distillate is highly pure water devoid of non-volatiles collected at room temperature. Reduced distillate conductivity estimates the quantitative reduction in salt concentration. Membrane distillation at lower temperature difference of 20°C

over ambient resulted in higher water flux and salt concentration at membrane interface of TS22 membrane as shown in Fig. 4.0. Cold feed water at 15°C is used at periodic interval to reduce scaling thereby lowering membrane maintenance costs.¹⁶ An intensification factor of 16 in terms of surface area requirements in comparison to conventional evaporation ponds for concentrated salt solutions has been possible. The above technique when integrated with low grade heat sources and environment tax benefits, can achieve high-quality water production at an optimized cost of US\$ 0.57 per cubic metre of pure water produced.¹⁶

Fig. 4.0: (a) Schematic process flow diagram of a typical membrane distillation unit (b) Variation of total solids content (TDS) in pre-filtered brackish water samples at feed side for different types of membrane material (PP22 and TS22) at temperature difference of 20°C¹⁶

5.3 High Gravity Processing

The application of high gravity field using a high-speed rotor allows counter-current gas-liquid flow through a rotating packed bed where one of the phases is fed continuously. It requires minimal area requirements, less maintenance, reduced equipment weight and provides short contact time over rotor packing with enhancement in heat and mass transfer rates.⁵ The need for de-aerated seawater in oil fields for re-injection purposes to avoid blocked pipe-works near oil-bearing rocks could be met through high-gravity processing (Higee) technique. Otherwise a conventional vacuum stripper of 3 m diameter and 10 m height with large auxiliaries would be required to reduce oxygen content to parts per billion (ppb) concentration level.¹⁷ To achieve effective momentum transfer to feed seawater (500 m³/hour), the requisite minimum pressure is of 2.2 bar for Higee deaeration operation. The energy requirements for Higee technology

primarily depends on the process time required for tip speed of rotor to be nearly equivalent to liquid velocity over radial direction. An intensification factor of 1000 in terms of oxygen concentration in treated seawater samples is estimated.¹⁷ Use of 400 RPM, 2.8 m length, 1.0 m diameter rotor with a cost-effective packing of reticulated metal foam reported a reduction of 90% in energy consumption. Major drawbacks in this ‘virtual’ separation column with evenly spread bubbles maintained throughout 10 mm thick packing are the resultant high pressure drop and backmixing problems.

Fig. 5.0: Schematic Diagram of Hige Unit

5.4 Spinning Disc Reactor

Spinning disc reactor (SDR) technology finds application where the problem of backmixing as in stirred tank reactors and higher energy requirements as in high-gravity field process reactors are eliminated with the utilization of thin liquid film formation specific to its spin disc design. It focuses on the intensification of spatial domain to obtain uniform mixing in laminar region (assumed) and very short residence time.^{5,18} A typical SDR design involves a liquid film of micrometer thickness over a spinning disc that is subject to high circumferential velocity (up to 1000 RPM) over ‘spiral grooves’ under an inert atmosphere, mostly nitrogen below 1 bar pressure.¹⁶ Coolants such as ethylene glycol or water are used to maintain disc operating temperatures. An example is a multiphase reaction for development of an alkyl cyanate ion intermediate (Darzen’s reaction) useful in drug development. It occurs within a second at 20°C with less than 0.1% impurity when compared to an operating temperature of 0°C with nearly 1.5% impurity in conventional batch reactors.¹⁸ Another reaction demonstrated in SDR is based on re-crystallization of like mesylate salt solution using hot acetic acid in tert-butyl methyl ether (an anti-solvent) to form an active pharmaceutical ingredient useful for various formulations.

PTFE coating over the spinning disc provides better control of particle size distribution with a maximum limit of 3 μm as illustrated in Fig. 6.0 (b) and prevent mass clusters.¹⁸ Some of the major limitations include control of turbulence of thin liquid film, process stability, maintaining vacuum conditions and material compatibility.

Fig. 6.0: (a) Operating principle of spinning disc reactor (SDR) (b) Comparison of particle size distribution between plant material obtained from conventional batch process and that from spinning disc reactor (SDR)¹⁸

5.5 Compact Heat Exchangers

Compact heat exchangers (CHE) provide increased available heat transfer area to exchanger volume thus lowering size and capital cost.⁵ Its values range between 300 to 700 m^2/m^3 for liquid phase and even higher for gas phase. Typical examples include plate or plate-fin heat exchangers and hybrid combination of reactors and heat exchangers fabricated on printed circuit boards for enhancement of heat transfer rates.¹⁹ This class of heat exchangers finds applications in heating, ventilation and cooling (HVAC) systems and heat pumps.¹⁹ During the last decade, two notable CHE designs evolved namely, Compabloc® from Alfa Laval, Sweden and 'Marbond' heat exchanger from Chart Marston, UK.^{1,19,20} Compabloc® is a laser-welded plate heat exchanger comprising corrugated plates with an alternate cross-flow pattern at high turbulence with multi-pass arrangement as shown in Fig. 8.0 (b). A reduced plant footprint is achieved by an intensification factor of 3.5 when compared to conventional shell-and-tube heat exchangers for the same thermal load. On industrial implementation, Compabloc® technology has been utilized for an evaporator unit in ammonium nitrate production plant in Slovakia from

2001 onwards that resulted in enhancement of heat transfer rates, lower fouling problems and a large reduction in energy consumption (~ 5 MW) for steam.²⁰ ‘Marbond’ heat exchanger, almost of the size of a ‘lap-top’ has been developed with hydraulic channels of sub-millimeter diameter and high voidage making it suitable for multiphase chemical systems. A significant reduction in capacity by about 95% can be achieved when compared to existing conventional shell-and-tube heat exchangers if corrosion-free environment is maintained.¹⁹ Some of the demerits of CHE technology include poor involvement of inertial forces, high pressure drop on use of highly viscous fluids and fouling problems.

Fig. 7.0: Comparison of different heat exchanger types in terms of hydraulic diameter (in mm) and area density (in m^2 per m^3)¹⁹. PHE: Plate heat exchanger, S+THX: Shell-and-tube heat exchanger, PFHE: Plate-fin heat exchanger, PCHE: Printed-circuit heat exchanger

Fig. 8.0: (a) Comparison of Alfa Laval's Compabloc® and conventional shell-and-tube heat exchangers for the same heat transfer duty (b) Section view of Compabloc® heat exchanger²⁰

6.0 Conclusion

Process intensification offers an effective approach in optimizing raw materials, land, utilities, energy input and capital costs resulting in efficient operating conditions for significant reduction in working capital and simultaneously maximizing output. The minimization of physical size of the plant by PI helps in better process control and sustenance of overall plant inventory.

The recent advances in PI concepts in chemical reactors, membrane separation and heat exchangers infer the quality of research work being carried out across the world. There is a need to address environmental issues associated with innovative PI technologies such as control over carbon footprint¹⁰, accumulation of unwanted by-products and their disposal methods. One of the feasible options could be the induction of renewable energy sources like solar, wind or bio-energy in PI technology applications to further offset CO₂ gas emissions and energy consumption. While addressing cleaner aspects of process technologies, PI strategies in reducing energy consumption (both temperature & pressure) and enhancing product yield would also improve safety concerns of the chemical industry.

In India, dedicated efforts on micro-reactors and membrane separation are underway at various Government funded academic institutions and research laboratories. On analyzing the energy use data, it is estimated that the Indian chemical sector could have reduced energy consumption by more than 20% in 2014 by adopting overall plant intensification in existing units.^{10,21,22} Presently, the major challenges exist in the commercial implementation of several PI technologies that have evolved over the past three decades. The primary reason hindering adoption of PI technology by industries could be attributed to an apprehension on delay over achievement of production targets accompanied by a risk of loss in market share. To ensure specific production requirements and smoother implementation of PI, industries need to partner with R&D or academic institutions at an early stage with investment in research to succeed commercially.

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