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The potentiality of process intensifying techniques for improvement of inherent safety in chemical processing

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ABSTRACT

Chemical industries are continuously faced with increasing challenges of safety requirements in plant design and operation. Consequently, more and more attention has been focused on developing greener, safer and efficient chemical processes employing process intensifying methodologies and equipment. While engineered safety devices can be added on to a plant as risk mitigation measures, safety is most reliably ensured by developing inherently safer techniques. This paper reviewed some of the process intensification approaches that could be utilized by chemical industries to improve inherent safety in plant design and operation protocols. Although the potentials of the techniques described in this review for intensification of chemical processing have already been proven in the laboratories, however their application on the industrial scale still presents a difficult challenge.

Introduction

The aim of every chemical industry is to run a plant that is profitable, safe, user friendly and environmentally benign so as to comply with legal requirements, protect company image and maintain economic competiveness. Over the years, the focus of the chemical and process industry has shifted towards the development of new technologies and innovations to advance inherent safety in chemical plant design and operating procedures [1].

The safety of a chemical process can be assured in two ways: (a) Inherent safety and (b) Engineered safety. The inherent safety is concern with the use of safer chemicals and operations. The essence of inherent safety is to avoid and remove hazards rather than to control them by the use of added-on protective systems referred to as engineered safety [2].

Process intensification (PI) is gaining much attention in both academia and industries as one of the key ideas in designing new plants and modifying existing units to improve inherent safety in chemical processing. It is a strategy for making drastic reduction in the size of a chemical plant by replacing large, expensive and energy-intensive equipment or processes with ones that are smaller, safer, less costly and more energy efficient [3]. A more expanded definition of PI is the development of novel apparatuses and techniques that brings about dramatic improvements in manufacturing and processing by substantially decreasing equipment-size/production-capacity ratio, energy consumption, or waste production, and ultimately resulting in cheaper, sustainable technologies [4].

According to Trvor Kletz, one of the pioneers of inherent safety concept, "What you don't have can't leak" [5], records have shown that industrial disasters involving complex processes or vessel of large volume usually fallout with high causalities, as evident in the industrial tragedies of Flixborough in 1974 [6] and Bhorpal chemical disaster in 1984 [7].

PI have been classified into process-intensifying methodologies such as new or hybrid separations, integration of reaction and separation, heat exchange, or phase transition and process-intensifying equipment, including novel reactors, intensive mixing, heat-transfer and mass-transfer devices [4].

This work present a review of potentiality of some process intensifying techniques that have been successfully applied for small scale chemical processes in the laboratories for improvement of inherent safety in chemical engineering processes.

Inherently Safer Methodologies for Process Intensification

Continuous flow chemistry

In recent years, continuous flow reactions on micro to meso scale have received considerable attention as a simple, efficient, and flexible platform for synthesis of various organic and inorganic molecules [8, 9]. The inherent closed nature of flow reactors enhanced reaction safety due to proper containment of harmful or toxic reagents and enable reactions to be carried out under superheated and/or pressurised conditions. The smaller reaction volume required in flow reactors in comparison to reactions in batch mode minimise the amount of chemical waste and allows reaction that are highly exothermic or hazardous to be run in a safer manner [10]. In addition, flow chemistry automation enable efficient control of reaction parameters such temperature, feed flow rate and reactant mole ratio quickly and easily during a reaction. By taking advantages of such features, various organic synthesis can be developed using continuous flow reactors.

FlowSyn reactor by uniqsis ltd is one of the commercially available continuous flow reactors currently employed for laboratory scale synthesis of various organic products [11]. The unit is an integrated instrument having a dual channel flow system, with each channel independently driven by a variable high-pressure pump. Gutmann et al. [12] reported a catalyst free continuous flow synthesis of tetrazole derivatives in a FlowSyn reactor. The procedure involves *in situ* generation of hydrozoic acid (HN_3) from sodium azide and acetic acid, followed by intensified high temperature and pressure addition to organic nitrile. It is worthy to note that *in situ* generation and consumption of HN_3 in the reactor, eliminates the need to store

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toxic, reactive and explosive intermediate and thus makes the continuous flow process greener and safer.

A continuous flow palladium catalysed alkoxycarbonylation of aryl halide developed by Kelly et al. [13] has shown considerable advantages in comparison to conventional batch mode using microwave heating. The flow chemistry approach has effectively contained the safety issues associated with loading significant quantity of toxic carbon monoxide (CO) into the reaction vessel which is the major challenge that arises in the batch process [14].

Due to the exothermic nature of nitration of aromatic compound especially on large scale, the batch method for this process is generally time consuming with lengthy and expensive safety assessments protocols. However, flow chemistry could provide a safer platform for nitration of aromatic compounds by providing a precise temperature control within the reactor. Moreover, the exothermic heat that is generated from small reaction volume in flow system can be rapidly dissipated without constituting any potential hazard. The Novartis Preparation Laboratories used Vapourtec R-Series flow reactor for nitration of 8-bromo-1H-quinolin-2-one, 2-amino-4-bromo-benzoic acid methyl ester and 1-benzosuberone in homogenous solutions [15]. The key to the success of this process is the employment of acetic and fuming nitric acid as nitration mixtures instead of traditional nitric and sulphuric acids mixture which is viscous and thus affects the flow rates of the reactants in the instruments. By adapting continuous flow process, not only was the handling of the exothermic reaction made easier, higher yield and selectivity was achieved in all the three nitration reactions compared to the batch process.

Several research groups have published interesting results for continuous flow synthesis with different organic reactions such as Hofmann rearrangement [16], alcohol oxidation [17] hydrogenation [18] and hydrolysis of ester [19] using self-designed flow chemistry apparatus or commercially available instrument.

Multifunctional reactors

Multifunctional reactive systems allow a unique way of achieving PI in the chemical and process industries. As the name implies, multifunctional reactors carry out one or more unit of operations simultaneously that would have been performed using separate equipment in the conventional process.

Reactive distillation (RD) is one of the emerging technologies that have extremely attractive potential as a process alternative for carrying out equilibrium limited liquid phase chemical reactions. It is a unit operation which combines chemical reaction and multi-component distillation in the same vessel in a single step [20]. RD technology has particular benefit for equilibrium limited reactions. By performing reaction and separation simultaneously, equilibrium can be shifted and in some applications almost complete conversion of the feedstock could be economically achieved [21]. RDC technology was successfully operated in commercial scale for the production of methyl acetate by Eastman chemical company [22].

The recovery of dilute acetic acid by esterification with n-butanol and iso-amyl alcohol in RDC has found to be a viable alternative to conventional methods such as azeotropic distillation, simple distillation and liquid–liquid extraction [23, 24]. RDC has been successfully employed for continuous epoxidation of alkene/terpenes with substantial benefits including increased selectivity, scalability and reproducibility in comparison with experiments carried out in a classical batch reactor [25].

A single-step *in situ* extraction and transesterification method known as reactive extraction have recorded remarkable successes in the production of biodiesel from crude palm oil [26], Jatropha curcas L. seeds [27] and castor seed [28]. In all these studies, the single step reactive extraction process achieved a higher yield of biodiesel as compared to other conventional method involving two separate processes of extraction and transesterification. Integration of chemical reaction and absorption of gases in liquid solutions has long been practiced in reactive absorption processes for treatment and purification of gases [29], removal of harmful substances [30] as well as in the production of fine chemicals [31].

Membrane reactor (MR) presents another type of multifunctional reactor that is capable of selective in-situ separation of product simultaneously with the reaction thereby shifting the equilibrium in favour of product formation [32]. In some cases, MR can be applied for a controlled feed distribution of a specific reagent in order to increase the overall yield of a process, avoid side reactions or catalyst deactivation [33]. Moreover, MR can be employed to regulate feed distribution in oxidative reactions involving hydrocarbons to ensure operation outside flammable region [34].

Membrane distillation

Hybrid separation mostly involves integration of membrane processes with one or more separation technique. Membrane distillation (MD) which is a separation process based on evaporation through pores of a hydrophobic membrane is perhaps one of the most widely known hybrid separation process. In MD process, the volatile component of a liquid feed stream diffuses through the pores of a membrane as a vapour, and condense on the other side of the membrane as a permeate liquid [35].

Ethanol is a known example of such substances that preferentially vaporize from aqueous solutions. Traditionally, ethanol is produced through batch fermentation process, which has low volumetric productivities and is time-consuming. However, MD have been described as a straightforward method of fermentation process, which leads to an increase in ethanol production by decreasing glycerol synthesis level and increasing the yeast cells number and viability [36].

Application of membrane separation in combination with reactive distillation have demonstrated bright potentials for processes including synthesis of n-propyl propionate from 1-propanol and propionic acid [37] and synthesis of ethyl tert-butyl ether from ethanol and tert-butyl alcohol [38].

MD could also be employed for sea water desalination, municipal and industrial waste water treatment. The temperature difference between two sides of the hydrophobic membrane results in difference in vapour pressure, so that waste water evaporates on the warmer side of the membrane and vapour diffuses through the pores to the cooler surface, where it condenses as purified water [39, 40]. There are currently water treatment plants utilising MD technology in the Florida Keys, Bahamas, and the Cayman Islands with capacity to purify 10,000 to 60,000 gallons per day [41].

Inherently Safer Equipment for Process Intensification

Catalytic monolith reactors

Monolith reactors are continuous unitary structures comprising several minute, parallel passages. They are widely used in hydrogenation [42] and environmental applications such as automotive exhaust gas cleaning [43]. The catalytic species are incorporated either into a thin layer of a porous oxide deposited on the channel wall (a wash coat), which acts as support for the catalysts or into the wall itself, to ensure

sufficient porosity and enhance the catalytic active surface [44]. Monolith reactor offers substantial benefits which include very low pressure drop along channels, high surface area per reactor volume, high catalytic efficiency and ease of separation of catalysts from reaction mixture.

Smits et al. [45] carried out assessment of the catalytic activity and selectivity of monolithic catalyst for three phase hydrogenation of mixtures of styrene and 1-octene in toluene. The simultaneous hydrogenation of styrene/1-octene mixture proceeds at high rates compare to trickle-bed reactor. Styrene was preferably hydrogenated to ethyl benzene while 1-octene was partially isomerised to internal olefins

A comparison among the monolithic and randomly packed reactors for the methanol conversion to propylene showed that the monolithic catalyst significantly enhanced the methanol conversion efficiency and the propylene selectivity [46]. The higher cell density and thinner wall of the monolith resulted in higher activity compared with the randomly packed catalyst pellets.

Microstructured reactors

Microreactors are chemical reactors of extremely small dimensions that usually have a sandwich-like structure consisting of a number of layers with micromachined channels [4].

The small channel sizes provide a high ratio of surface area to volume, which leads to efficient heat and mass transfer characteristics. Another benefit of microreactors is the ability to safely carry out reactions in the explosive regime, which can open up new reaction pathways or can avoid the use of large dilution streams [47].

A microreactor containing platinum nanoparticles catalyst has been applied for hydrogenation of nitrobenzene to aniline [48]. Compared with the conventional carbon supported platinum nanoparticles (Pt/C) used in batch experiments, the nanoparticles inside the microreactor exhibited higher catalytic activity and were easily regenerated after they were deactivated.

Centrifugal fields

The application of centrifugal fields such as rotating devices for enhancement of chemical reaction, separations and heat and mass transfer is one of the most remarkable innovations that could potentially be adopted for intensification of chemical processes, especially for multiphase systems [49]. Spinning disc reactors (SDC) are some of the widely known rotating devices that have become particularly attractive in chemical engineering applications due to their excellent mass and heat transfer and uniform micro-mixing characteristics [50]. In SDC, the disc surface generates a high centrifugal force resulting in dispersion of liquid as a thin film which gets in contact with a gas [51]. The gas could either be reactive or inert and could be employed as a coolant or heating medium. Application of a thin film reactor using SDR technology have achieved significant reduction in processing time for the continuous free radical polymerisation of styrene [52].

Rotor-stator mixers are currently receiving much attention and have found application in the food, pharmaceutical and cosmetic industries for processing of colloidal liquid-liquid systems and for dispersing or breaking solid particles and aggregates [53]. Rotor-stator mixers are characterized by small gap between the rotor and the stator and could achieve PI by incorporating several process operations within a single unit operation [54]. The main advantage of rotor-stator is their ability to create high shear rates in the rotor–stator gap and high energy dissipation rates since the kinetic energy generated by the rotor is dissipated in the small stator volume [55]. A notable industrial implementation of rotor-stator mixers is the Jet impingement reactor developed by Noram Engineering and construction [56] for liquid-liquid reactions. The invention is generally applicable for immiscible liquid-liquid reactants and particularly suitable in the nitration of aromatic hydrocarbons using mixed acids in aqueous solutions.

Conclusion

Process intensification is an important strategy in the development of inherently safer chemical processing. By reducing the volume of hazardous material or replacing large, energy intensive equipment and processes with the ones that are smaller, less costly and energy efficient, the safety and efficiency is greatly improved. Moreover, significant savings in investments, raw materials and energy cost is achieved.

PI enables chemical processing to be carried out with optimum conditions thereby achieving maximum yields and product selectivity in shorter time. The overall benefits of PI have positive effects on the environment, health safety and production economics. Rather than relying on engineered safety devices and other consequence mitigation systems, the safety of a chemical plant is more reliably assured by minimizing the likelihood of any possible hazard.

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