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Review Paper

Process intensification in gas/liquid/solid reaction in trickle bed reactors: A review

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ABSTRACT

As an important form of reactors for gas/liquid/solid catalytic reaction, trickle bed reactors (TBRs) are widely applied in petroleum industry, biochemical, fine chemical and pharmaceutical industries because of their flexibility, simplicity of operation and high throughput. However, TBRs also show inefficient production and hot pots caused by non-uniform fluid distribution and incomplete wetting of the catalyst, which limit their further application in chemical industry. Also, process intensification in TBRs is necessary as the decrease in quality of processed crude oil, caused by increased exploitation depths, and more restrictive environmental regulations and emission standards for industry, caused by increased environment protection consciousness. In recent years, lots of strategies for process intensification in TBRs have been proposed to improve reaction performance to meet the current and future demands of chemical industry from the environmental and economic perspective. This article summarizes the recent progress in techniques for intensifying gas/liquid/solid reaction in TBRs and application of intensified TBRs in petroleum industry.

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1. Introduction

Gas/liquid/solid multiphase reaction plays an important role in chemical reaction engineering and has been regarded as one of main conference topics in important international conferences, such as International Symposia on Chemical Reaction Engineering (ISCRE) and International Symposia on Catalysis in Multiphase Reactors (ISCMR). As an most important type of multiphase reactors, trickle bed reactors (TBRs) show advantage in flexibility, simplicity of operation, large annual throughputs and are extensively applied in chemistry industry, including petroleum refining (Yuan et al., 2020; Behnejad et al., 2019; Kaiser, 2017; Ghassabzadeh et al., 2020; Liu et al., 2011; Chi et al., 2013), petrochemical industry (Dashliborun et al., 2016; Gorshkova et al., 2012), biochemical engineering (Devarapalli et al., 2016; Karimi et al., 2009), environmental engineering (Rachbauer et al., 2016; Üresin et al., 2015; Kim and Ihm, 2011; Mudliar et al., 2010), electrochemistry (Abdullah and Xing, 2017) and fine chemical industry and pharmaceutical industry (Bonrath, 2014; Zhang et al., 2015), with the targets of conversing unsaturated organic compounds into saturated products, upgrading petroleum raw materials and intermediates as well as conversing undesirable by-products into environmentally acceptable and recyclable products (Fig. 1). In recent years, requirements of stringent environmental laws and regulations have necessitated the in-depth processing. For example, in deep hydrodesulfurization of diesel fractions, TBRs become a potential type of reactors, which remove a few parts per million of refractory sulfides.

TBRs are employed for gas-liquid-solid reactions, in which liquid and gas phases flow (either countercurrently or concurrently) through a fixed bed of catalyst particles, where reactions take place (Fig. 2). Plug flow is nearly achieved in TBRs because of the motionless catalyst bed, and in this regard, they are superior to other gas/liquid/solid three-phase reactors where catalyst is either fluidized or slurried. TBRs show high catalyst loading per unit volume of liquid and low energy dissipation rate and thus have the

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Fig. 1. Application examples of TBR.



Fig. 2. Schematics of TBRs. (a) Cocurrent downflow; (b) cocurrent upflow; (c) Countercurrent flow.

advantage of massive production compared with slurry reactors. Based on different gas and liquid flow rates, fluid properties and bed characteristics, four flow regimes exist: trickle flow, pulse flow, spray flow and bubble flow. Trickle flow, characterized as small liquid flow rates and low or moderate gas flow rates, is the most routine operation regime applied in industrial processes. However, drawbacks in TBRs, such as uneven gas-liquid distribution and low catalyst wettability, cause uncontrollable reaction process, lower yield, more by-products and hot spots for highly exothermic reaction and thus lead to the limitation in their application. Enhancement in reaction performance of TBRs is necessary for their efficient application and for meeting the requirements of green chemistry in chemical industry. Moreover, any advancement of TBRs, even if is very small, brings considerable cost saving and economic benefits.

Process intensification techniques, characterized by significantly improving process efficiency, that is, reducing energy consumption, by-product formation and equipment size, considerably contribute to sustainable development in chemical engineering. Methods of improving reaction performance in TBRs through process intensification (Fig. 3) have drawn great attention from researchers.

In this article, strategies and mechanisms in process intensification of gas/liquid/solid catalytic reaction in TBRs over the last decade from different methods are summarized, including operating methods and techniques, solvent, catalyst, reactor structure as well as alternative energy sources, and their advantages as well as disadvantages are discussed accordingly.



Fig. 3. Process intensification techniques. Reprint permission obtained from Stankiewicz (2003).

2. Process intensification techniques

A series of mass transfer and reaction processes (Fig. 4) are involved in gas/liquid/solid three-phase reaction in TBRs. Reaction rate is restricted by hydrodynamics, mass and heat transfer composed of gas-to-liquid (Tadepalli et al., 2007), liquid-to-solid and inside solid phase steps and intrinsic kinetics, which are influenced by operating parameters, liquid holdup, wetting efficiency, intraparticle diffusion, axial dispersion and catalyst-bed structure. Conventional techniques in enhancing reaction performance in TBRs are mainly realized by increasing temperature to enhance catalytic activity, increasing operating pressure to improve gas solubility or increasing concentrations of reactants, catalysts and co-catalysts within the operating ranges. In this article, process intensification by applying novel operating methods, solvent, catalyst, reactor structure and alternative energy sources are mainly discussed.

2.1. Operating methods and techniques design

TBRs are usually operated at low gas/liquid flow rates, in which interaction between gas and liquid is quite poor. Researchers have been trying to improve catalytic performance by adopting novel operating methods and techniques, taking cycle and upflow modes as typical examples, which are both effective for increasing fluid distribution uniformity and improving wetting.

unsteady state operation mode, in which reaction performance is enhanced by periodically changing operating conditions such as reaction temperature, gas flow rate, liquid flow rate, reactant concentration and catalyst activity, by improving the competition between the gas and liquid phases in supplying reactants to reactors, as well as the complex interactions between hydrodynamics and reaction. In this mode, catalyst surface is renewed periodically and interaction between gas and liquid phases is enhanced, thereby effectively improving both wetting and flow distribution (Dashliborun et al., 2016). Dietrich et al. (2012) proposed a reliable periodically operating effective assessment method, in which improved local liquid distribution is demonstrated by visualization technology of Nuclear Magnetic Resonance tomography (NMR-tomography). Hamidipour et al. (2013) successfully predicted the hydrodynamics of TBRs by Computational Fluid Dynamics (CFD) simulations and verified the model by Electrical Capacitance Tomography (ECT) imaging under different cyclic operation conditions. Wongkia et al. (2013) demonstrated increased conversion in TBRs under unsteady operation. Although the merits of unsteady operation praised in the literature as one of the process intensification strategies advocated for TBRs, there is still reluctance to implement it in industrial. It can partly be ascribed to the lack of hydrodynamics studies relevant to complex nonlinear behavior of cyclic operating mode (Atta et al., 2014), engineering data relevant to the elevated temperature and pressure characterizing industrial

Cycle mode (Matthischke et al., 2018; Urmès et al., 2020) is an



Fig. 4. Determination of reaction control steps.

processes and research relevant to the effect of reactor scaling-up on periodic operation performance. Therefore, more flow visualization should be directed to hydrodynamics studies for different operations. More unsteady state CFD models need to be developed involving all possible parameters for extensive research, which lay the foundations for industrial scale-up.

Upflow provides better axial and radial mixing than downflow mode, with significant advantages in terms of liquid distribution, selectivity, temperature control and scaling-up. Moreover, increase in liquid holdup as well as long residence time eliminates generation of hot spots. Wu et al. (1996) systematically analyzed the research results under different operating modes and proposed reasonable approaches to adapt different operating modes. Houwelingen and Nicol (2011) found a greater conversion in upflow within trickle flow operation than downflow. There are also deficiencies in upflow, mainly includes increased liquid film thickness and pressure, both caused by increased holdup. The former one causes increased external mass transfer resistance from gas phase to catalyst surface, when the latter one leads to thermodynamic instability.

As the strong interaction between gas and liquid phases and 4 times higher mass transfer rate compared to trickle regime in pulse regime, pulse flow operation mode has become a new enhancement method. Inclined rotating tubular fixed bed reactor (Dashliborun et al., 2017) is considered to be a new and effective pulse flow regulation method, which can effectively suppress hot spots because of the periodic renewal of liquid on catalyst surface, extend catalyst lifetime because of uniform utilization of catalyst bed and flexibly adjust liquid residence time by controlling the tilt angle and rotation speed. Härting et al. (2015a, b, c) showed that reaction performance in the above novel reactor increased by 2 times in comparison to that in traditional TBRs.

In addition, reactor performance is also influenced by factors

such as elevated-temperature and high-pressure conditions, uniqueness of bed structure, complexity of reaction and hydrodynamics phenomena. Therefore, studies have been carried out to understand and quantify hydrodynamics of TBRs. Advanced techniques such as computed tomography (CT) (Johnson et al., 2017), wire mesh sensor (WMS) (Schubert et al., 2010), magnetic resonance imaging (MRI) electrical resistance tomography (ECT) (Hamidipour et al., 2019), electrical capacitance tomography (ECT) (Hamidipour et al., 2009) and X-ray digital industrial radiography technique (DIR) (Salleh et al., 2014) have been used to obtain internal structure of catalyst bed and gas-liquid distribution under actual operating conditions. The combination of the above advanced techniques and computational fluid dynamics (CFD) provides a powerful tool for studying fluid flow phenomena as an alternative to empirical models.

In the current study, most TBR simulation (Fig. 5) focus on mass and heat transfer model, multiphase flow model, porous media model and drag force model. In most cases, reactions in TBRs are limited by mass transfer because of poor interaction between gas and liquid phases and low mass transfer rate compared to other heterogeneous catalytic reactors with intense mixing process. Therefore, developing mass transfer models is necessary for better understanding TBRs (Table 1). Also, establishing suitable heat transfer models and accurately estimating heat transfer rate are important in reactor design and scale-up for avoiding thermal runaway caused by low heat transfer rate and a large amount of heat released by reactions. Mass and heat transfer are affected by momentum transfer involving flow velocity and distribution of gas and liquid phases, which makes it important to study momentum transfer model. Among them, multiphase flow model includes Euler-Lagrange and Euler-Euler methods, the former of which is applied to discrete phase and the latter is applied to continuous phase. The Euler-Euler method is the most widely used in the



Fig. 5. Numerical simulation of TBRs in literature.

Table 1

Mass transfer correlations used in some systems.

System	Correlations	Conditions			Reference		
		<i>Т</i> , К	P, MPa	$u_G/Q_{m,G}$ /SV	$u_{\rm L}/Q_{m,{\rm L}}$	Catalyst	
Hydrogenation of α- methylstyrene	$\frac{Sh}{Sc^{0.33}} = 0.395 Re_{\rm eq}^{0.64}$	413.15	1	0.35 m/s	$\begin{array}{l} 3.2 \times 10^{-2}\text{-} \\ 6.4 \times 10^{-2} \text{ g/s} \end{array}$	Pd/γ- Al ₂ O ₃	Shigarov and Kirillov (2009)
	$\frac{Sh}{Sc^{0.33}} = \frac{0.468Re_{\rm eq}}{1.18Re_{\rm eq}^{0.41} - 1.52}$						
	$\frac{Sh}{Sc^{0.33}} = 0.201 Re_{\rm eq}^{0.707}$						
Hydrogenation of benzene	$k_{\rm LG}a \propto \frac{u_{\rm GS}}{u_{\rm LS}}$	343	1.6	5.98×10^{-9} - $1.12 \times 10^{-7} \text{ m}^3/\text{s}$	$1.5 imes 10^{-6}$ - 3 imes 10^{-6} kg/s	Ni/ Al ₂ O3	Kallinikos and Papayannakos (2010)
Hydrogenation of octenes and isooctenes	$k_{\rm LS}a \cdot f = \frac{k_{\rm R1} - k_{\rm R2}}{k_{\rm R1}/k_{\rm T1} - k_{\rm R2}/k_{\rm T2}}$	333.15	5	1×10^{-5} - 2 × 10 ⁻³ m/s	1.8×10^{-3} - 7.5×10^{-3} m/s	Pd/γ - Al ₂ O ₃	Houwelingen and Nicol (2011)
Hydrogenation of	$Sh = 2 + 54.7Sc^{1/3}We^{1/2}\varphi^{1/10}$	303.15	0.1	5.67×10^{-3}	6.37×10^{-4}	Pd/	Tan et al. (2012)
Ethylanthraquinone	$\varphi = Q_{\rm G}/(Q_{\rm G}+Q_{\rm L})$	-353.15	-0.4	$2.83 \times 10^{-2} \text{ m/s}$	$6.37 \times 10^{-3} \text{ m/s}$	AI_2O_3	
Hydrogenation of α- methylstyrene	$Sh = 801 \left(\frac{u_{\rm G,S}}{[\rm m \cdot s^{-1}]} \right)^{0.24} \left(\frac{u_{\rm L,S}}{[\rm m \cdot s^{-1}]} \right)^{0.49}$	343.15 373.15	1.1	0.002–1.18 m/s	5×10^{-3} - 10^{-2} m/s	Pd/γ- Al ₂ O ₃	Langsch and Haase (2013)
Catalytic wet air oxidation o	$f Sh = 2.29\phi^{-0.91}(Re_1 + Re_c)^{0.04}$	373.15	0.6	3×10^{-3} -	5×10^{-4} -	Pd/	Tan et al. (2019)
phenol	···· -···· (···· [· ··· G)	-453.15	-1	$2.5 \times 10^{-2} \text{ m/s}$	$4 \times 10^{-3} \text{ m/s}$	Al_2O_3	
	$\phi = \frac{d_{\rm i}}{d_{\rm p}} C a_{\rm L}^{-0.19} \left[\left(\frac{u_{\rm G, sup} + u_{\rm L, sup}}{u_{\rm G, sup}} \right)^{1/3} - 1 \right]$						

simulation of TBRs, which includes Eulerian model, volume of fluid model and mixture model. The Eulerian model (Jindal and Buwa, 2017; Jejurkar et al., 2020), which was developed to simulate homogeneous and heterogeneous reactions by taking account interaction among phases, is most widely applied in TBRs simulations. The VOF model (Deng et al., 2020) mainly focuses on capturing characteristics of phase interfaces. The Mixture Model is applicable to bubble flow. The porous media models (Lu et al., 2018; Mohammadpour et al., 2019) were developed to simulate catalyst particles in TBRs, in which establishment of complex geometric structures can be avoided. In addition to multiphase flow model and porous media model, the drag force model is also important for the simulation of TBRs. The relative permeability model (Xiao et al., 2012), in which the concept of relative permeability was introduced to modify the drag force expression for single phase flow, is applicable for two-phases flows, although without taking into account of gas-liquid interaction forces. The silt model (Iliuta and Larachi, 2009), in which fluid flow around catalyst bed is regarded as flowing through a rectangular slit, is applicable to poor interaction condition by ignoring gas-liquid interaction and taking into account of slip effect. The fluid-fluid interfacial force model (Hamidipour et al., 2013), in which gas-liquid interaction are considered, can reasonably predict pressure drop and liquid holdup under high pressure or high gas concentration.

2.2. Solvent design

During the reaction, solvent influences reactant solubility, catalyst dispersibility, adsorption and desorption behavior of catalyst surface, and it also acts as a heat carrier for heat released in exothermic reaction as well as heat supply in endothermic reaction. Organic solvents, with great solubility, help enhance reaction rate in gas/liquid/solid three-phase catalytic reaction processes (Wu et al., 2017) and are widely used in chemical production. In most cases, alcohol is used as the solvent in hydrogenation reaction to improve conversion and selectivity (Table 2). Some new reaction solvents are employed for process intensification in hydrogenation reaction. For instance, Bai et al. (2019) developed a solvent mixture consisting of trimethylbenzene and water-insoluble organic heterocyclic bisamide for enhancing hydrogenation efficiency of 2alkylanthraquinone. Application of organic solvents significantly increases cost and shows adverse effect on human health and environment. In recent years, innovative solvent systems have been developed for higher reaction productivity and lower environmental impact, mainly including supercritical fluids (SCFs) and ionic liquids (ILs).

SCF, as a "green" alternative to traditional organic solvents, has become more and more impactive in heterogeneous catalytic hydrogenation reaction because of its high density, high diffusivity and low kinematic viscosity (Seki et al., 2008). Also, SCF shows precise control of reaction environment and regulates the selectivity (Gadge and Bhanage, 2016). Among all SCFs, supercritical carbon dioxide has received widespread attention because of its low cost, non-toxicity, non-flammability, green, easy disposing and recycling. Catalytic hydrogenation reaction, which is diffusionlimited in traditional organic solvents, can be carried out in supercritical CO₂. More and Yadav (2018) proved that application of SCF significantly increases the hydrogen concentration on catalyst surface, leading to a higher reaction rate.

ILs, composed of organic cations and organic or inorganic anions, are gradually replacing traditional organic solvents in catalysis, petrochemical industry and other chemical engineering industries. ILs have received widespread attention because of their low-cost, non-toxicity, negligible vapor pressure, high thermal stability, high solubilizing ability for organic and inorganic compounds and excellent designability according to needs (Garg et al., 2020).

2.3. Catalyst design

Catalysts vary reaction rates within 3–6 magnitude and also play an important role in improving overall performance of heterogeneous catalytic process. Catalyst design has become one of the most important strategies to enhance catalyst activity (Comandella et al., 2017), prolong catalyst lifetime (Davie et al., 2008), increase catalyst mechanical strength (Zhao et al., 2019), improve product selectivity, reduce costs, decrease pressure drop and realize uniform liquid distribution (Honda et al., 2014). This section presents an overview of recent work in catalyst design, mainly including the shape, size, mechanical strength, main active constituents and filling method.

The shape, size and mechanical strength of catalyst particles play significant roles in catalyst design for minimizing pressure drop and maximizing catalytic activity. Using irregular catalysts normally results in under-utilizing active constituents and high pressure drop. Thus, commercial catalysts are commonly utilized in various physical forms such as pellets, granules and extrudates. Cao et al. (2016) established correlations of shape-dependent structural and chemical factors of catalyst with catalytic activity and selectivity. Quintana-Solórzano et al. (2018) refitted previous pressure drop correlations (Harrison et al., 2013) to better describe hydrodynamics of reactors packed with different particles configurations. Particleresolved CFD simulations (Karthik and Buwa, 2020), linking particle-shape effects to reactor performance, have become a special design method for developing optimal catalyst shapes and been validated against experimental results or correlations concerning pressure drop and bed voidage (Wehinger et al., 2017). Most validation studies performed were conducted on simple particle shapes, including spheres, Raschig rings and cylinders. Jurtz et al. (2020) simulated hydrodynamics in the packed-bed filled with different complex catalyst shapes by applying particle-resolved CFD and validated against experimental results. The reduction of catalyst size leads to the increase in effective contact area, which improves catalyst activity. Nano-sized catalyst particles exhibit relatively higher catalytic activity compared to micron-size or milli-size particles for the same mass catalyst (Intanin et al., 2020) because of increased specific surface area. For example, hybrid nanoparticles (Awad et al., 2020), combining inorganic and organic components on the molecular scale, provide a new idea for obtaining catalysts with excellent performance in catalytic hydrogenation; magnetic recyclable nanocatalysts (Jiang et al., 2017) show excellent catalytic activity and reusability in catalytic hydrogenation of styrene and 4nitrophenol. Low mechanical strength in TBRs causes large pressure drop and uneven gas/liquid distribution, leading to low catalytic efficiency or even plant shutdown, and, thus, improving mechanical strength is important. Powder processing parameters (Zhao et al., 2019), mainly including binder and water amount as well as pH of solvent, are frequently optimized for improving mechanical

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Literature summary on solvent effect in hydrogenation reactions.

Reaction system	Effect of solvent on reaction rate	Effect of solvent on selectivity	Catalyst	Reference
Hydrogenation of cyclohexene	Reaction rate ∝ Molar fraction of room temperature ionic liquid	Selectivity \propto Molar fraction of room temperature ionic liquid	Pt/ Al ₂ O ₃	Khodadadi- Moghaddam et al. (2009)
Hydrogenation of 2- methyl-3-butyn-2- ol	i-PrOH > EtOH > H ₂ O + EtOH	Toluene > hexane > heptane > octane > cyclohexane	Pd/ MN270	Nikoshvili et al. (2015)
Hydrogenation of butyronitrile	Methanol > benzene > toluene > cyclohexane	Methanol > benzene > toluene > cyclohexane	Co/SiO ₂	Segobia et al. (2015)
Hydrogenation of 4-	Alkanes, secondary alcohols > primary	Cyclohydrogenation: alkanes;	Pt/TiO ₂	Mcmanus et al.
phenyl-2-butanone	alcohols	Carbonylhydrogenation: aromatics and alcohols		(2015)
Hydrogenation of	Aprotic > Protic \gg Apolar solvents	Aprotic polar solvents (excluded tetrahydrofuran): COL (60%);	Pd/C	Li et al. (2018c)
cinnamaldehvde		Aprotic apolar (excluded benzene), protic: HCAL (80% or more)		

stability. 3D printing, fabricating the designed product by obtaining a virtual design from computer-aided design (CAD) software and printing layer by layer, provides a novel technique to maximize the catalytic surface and adjust manufacturing parameters for obtaining excellent mechanical properties.

Precious metals, such as palladium, platinum and rhodium are frequently applied as catalyst in heterogeneous catalytic reaction in TBRs to achieve higher conversion under mild operating conditions. Large-scale consumption of precious metals may raise concerns about sustainability of economic and environmental, and, thus, development of low-cost catalysts is important. Sponge metals, such as Raney Co (Ra-Co) and Raney Ni (Ra-Ni), have been reported as potential catalysts (Loos et al., 2016) for continuous catalytic reaction because of their high porosity and mechanical properties, low cost and pressure drop (Islam et al., 2019). Said et al. (2017) demonstrated high selectivity of Ra–Co catalyst in catalytic hydrogenation of iodo-nitroaromatic refametinib active intermediates in TBRs. Carangio et al. (2020) demonstrated high conversion of Ra-Ni in the reduction of aliphatic nitro and without any obvious deactivation during continued operation. However, there is still safety issues need to be solved in the operation of sponge metal (Jindal and Buwa, 2017). In addition, combining a small amount of precious metals and relatively inexpensive components is promising for reducing costs and improving catalytic efficiency. In general, compared to monometallic metal nanoparticle catalysts, bimetallic metal nanoparticles show higher catalytic activity (Fulajtárova et al., 2015). Jiang and Xu (2011) reviewed the latest developments in multimetallic nanoparticles for improving catalytic performance. Shesterkina et al. (2016) reported the increase in catalytic activity and selectivity in hydrogenation of phenylacetylene to styrene by applying Pd-Fe/SiO₂ bimetallic catalyst compared with Pd/SiO₂ catalyst.

Carbon, γ -aluminum oxide, silica, zeolite, polymer, gel and glass fiber are frequently applied as support in heterogeneous catalytic reaction in TBRs. For Preventing the aggregation of nanoparticles, ligands (Moghaddam et al., 2018), ILs (Salminen et al., 2014), surfactants (Harraz et al., 2012), dendrimers (Bingwa and Meijboom, 2015), polymer (Comandella et al., 2017), periodic open cellular structure (POCS) (Lämmermann et al., 2018), metal-organic frameworks (MOFs) (Isaeva et al., 2011) and microgel (Tan et al., 2016) have been developed to stabilize nanoparticles and improve catalyst stability. For example, Harraz et al. (2012) reported Pd/PEG catalyst, which exhibits excellent stability, catalytic activity and selectivity for hydrogenation of styrene under mild operating conditions in an environmentally friendly solvent system. Zhang et al. (2014) and Li et al. (2016) reported novel MOFs catalyst, encapsulated with precious metal nanoparticle, can significantly improve the stability and selectivity of the product. Comandella et al. (2017) reviewed various methods for embedding palladium catalysts through polymer films to avoid catalyst deactivation. Yousaf et al. (2017) developed a unique structure by loading palladium nanoparticles on MnO_x-CeO₂ mixed oxides for catalytic hydrogenation. In addition, many new carbon materials are also applied as catalyst supports, including fullerene (Qu and Chen, 2020), graphene (Wei et al., 2016) and mesoporous carbon (Feitosa et al., 2019). In many cases, "bifunctional catalysts" (Jin et al., 2016) are applied to increase product selectivity.

Industrial TBRs are packed by the sock packing method, in which particles are randomly introduced inside the packed bed, or the dense packing method. In the latter one the number of contact points and pressure drop significantly increase. Many new catalyst filling methods and packings have been proposed to improve fluid distribution of the gas-liquid mixture on catalyst surface and increase gasliquid contact efficiency as well as mass transfer rate. For example, monolithic catalysts (Tsai and Ma, 2018), open-celled foams (Nie et al., 2017), knitted wire packing (Wen et al., 2020), corrugated sheet packings (Singh et al., 2017) and winpak (Qi et al., 2017). These new structured packings have been proved to be feasible on the laboratory scale because of high porosity, high geometric surface area, low pressure drop and better heat transfer characteristics. However, structured packings are still not yet commercially available because of the low catalyst loading and high cost.

The full utilization of catalyst, especially in the trickle flow regime, relies on well-dispersed liquid feed on catalyst bed, which means both desired bed loading and effective liquid inlet distributors are necessary. In downflow operation mode of packed beds, both gas and liquid enter reactor from the top through a mixer, then pass through pre-distributor and redistribute through the second or principal distributor tray. Mixed liquid and gas phases are evenly sprayed onto the catalyst bed by distributors. Distribution effect of distributors directly affects apparent reaction rate, product yield and catalyst deactivation rate in reaction process. Especially, when most highly exothermic processes are carried out in TBRs, inhomogeneous distribution of gas and liquid phases could be amplified because of fast drying of catalyst. At present, there are many types of distributors applied in industrial TBRs, mainly including bubble cap trays, jet distributors, perforated plate or sieve trays, chimney trays and gas assisted lift tube trays. Distributor performance could be measured by its liquid discharge pattern, liquid distribution, pressure drop, coverage span, fouling surfaces and sensitivity to tray levelness (Jain et al., 2020). Bazer-Bachi et al. (2013) gave some guidelines for choosing distributors by comparing intrinsic performance of distribution and resistance to trav unlevelness in different distributors. Jain et al. (2020) developed a Eulerian twofluid model to simulate gas-liquid flow in distributors and validated it by applying high speed imagine, voidage probes and pressure drop measurements. Lopes et al. (2018) developed three novel flow distributors, evaluated their performance by varying the distributor height as well as the presence or absence of internal obstacle and analyzed the phase distribution uniformity.

2.4. Novel reactor structure design

Most heterogeneous reaction in TBRs is limited by external mass transfer and heat transfer. In addition, difficulties with heat dissipation may lead to hot spots resulting in catalyst deactivation. Therefore, some new reactor structure has been proposed to improve the contact between phases and increase mass and heat transfer rate.

2.4.1. Microreactors

Application of micro-structured devices intensifies mass and heat transfer of gas/liquid/solid catalytic reaction by reducing diffusion time and increasing mass transfer area. They have become one of the significant methods of chemical process intensification because of their high specific surface area, safety, waste minimization and precise control of reaction temperature. As shown in Fig. 6, microreactors mainly include micro-packed bed reactors, chip-based microreactors, catalytic membrane reactors, monolithic microreactors, polytetrafluoroethylene capillary microreactors, falling film microreactors and so on.

Micro-packed bed reactors (MPBRs) show increased product selectivity (Zhang et al., 2017) and faster reaction rate, and varies mass transfer coefficient within 2 magnitudes (Sang et al., 2020), achieving continuous flow under fast dynamic control (Yang et al., 2018). Most well-known processes in chemical industry, such as hydrogenation, HDS and Fischer-Tropsch synthesis, are carried out in MPBRs. There are some shortcomings in MPBRs, mainly include poor reproducibility caused by inherent randomness of catalyst distribution (Navarro-Brull and Gómez, 2018) and high pressure



Fig. 6. A variety of microreactors. Reprint permission obtained from Expósito et al. (2019), Li et al. (2018a, b), Liu et al. (2018, 2020), Rehm et al. (2016), Sun et al. (2018), Tan et al. (2019), Truter et al. (2016).

drop caused by small catalyst particles. Different designs and configurations (Faridkhou et al., 2016) are designed for reducing pressure drop, such as a radial design and a cross-flow design. In addition, expanding the scale of microreactor to millimeter reactor (Piskun et al., 2016) has also been proposed to reduce pressure drop. Catalytic wall reactors (Moreno-Marrodan et al., 2018), with a thin layer of catalyst coated on the inner wall, have also been developed to reduce random distribution of catalysts.

Chip-based microreactors (Expósito et al., 2019), with unique advantages of low reactant consumption, high sensitivity, high mass transfer rate and environmental friendliness, have received widespread attention. For instance, Truter et al. (2016) demonstrated improved residence time control and increased yields obtained in microchannel reactor. Being able to combine with microscopic visualization is another significant advantage of chipbased microreactors, which makes reaction and mass transfer phenomena more understandable. Micro-structured devices also help enhance chaotic convection (Martínez et al., 2017), among which embedding obstacles (Pordanjani et al., 2018) in the chipbased microreactors is simple and most widely studied methods. However, application of the microchannel reactors is still limited in heterogeneous catalysis because of the lack of suitable methods for loading various catalysts in microfluidic devices, controllability of catalyst position and homogeneous distribution of catalyst active

sites (Xu et al., 2013).

Catalytic membrane reactors (CMRs), combining membrane separation and catalytic reaction, have become an effective way to improve fluid distribution, mass and heat transfer (Table 3). In CMRs, membrane could be used either as a catalytic active material, such as palladium membranes or as a catalyst support, such as palladium nanoparticle-loaded microporous ceramic. The transfer of reactant components to reaction zone can be controlled by membrane to strengthen gas-liquid mass transfer (Aran. 2011). improve product selectivity (Escolástico et al., 2016) and improve system security of premixed explosion systems. CMRs show good application prospects because of the numerous advantages over conventional catalytic and separation process. There are also some shortcomings still need further studied, mainly including the adsorption and diffusion of reactants, solvent and product on the membrane, lack of appropriate modification methods to obtain the required distribution of active catalyst components, deactivation of catalyst and blockage of membrane pores.

Novel reactor structures, coupling gas-liquid microdispersion technique (Ji et al., 2020) (Fig. 7) with TBR, were developed to increase processing capacity and address difficulty in processing of microreactors, expanding their application in industrial processes with high throughput. Tan et al. (2012) proved that the mass transfer coefficient in this novel reactor structure is two orders of

Table 3

Some examples of CMRs applied in catalytic reactions.

Membrane	Method	Activity testing	Reference
Porous expanded polytetrafluoroethylene (ePTFE) membrane	Deposited Ru catalyst particles	Aqueous phase hydrogenation of levulinic acid	Stanford et al. (2016)
Porous ceramic alumina (α -Al ₂ O ₃) hollow fiber	Nickel deposition-precipitation	Hydrogenation of nitrite	Espinosa et al. (2018)
Hollow fiber membrane made of corundum (α -	Sputtering, microemulsion, impregnation,	Situ epoxidation of trans-chalcone and phenol	Pinos-Vélez et al.
$Al_2O_3)$	precipitation of Fe complex	peroxidation	(2019)
Commercial microfiltration ceramic membrane	Impregnation	Hydrogenation of 1-butyne	Colli et al. (2019)
Perovskite hollow fiber membrane	Conventional solid-state reaction	Partial oxidation of methane	Jiang et al. (2020)



Fig. 7. A variety of micro-dispersion system. Reprint permission obtained from Tan et al. (2012), Tan et al. (2019), Ji et al. (2020).

magnitude higher than that of traditional TBRs by applying stainless steel microfiltration membrane as the dispersion medium. Our previous studies (Tan et al., 2019) have shown that disappearance rate of phenol in catalytic wet oxidation of phenol increased by 70% by applying micro-dispersion device.

Monolithic microreactors (Maresz et al., 2020), integrating the active phase into a straight channel or a macroporous monolithic structure, were developed to eliminate hot spots and stagnation areas. Studies (Song et al., 2016; Chen et al., 2018) demonstrated the increased controllability of operating conditions, conversion and selectivity in above novel microreactor.

2.4.2. Catalytic static mixers (CSMs)

CSMs (Hornung et al., 2017), constructed by retrofitting the tubular reactor with specially designed catalytic insert, were developed to achieve effective mixing of reactants, heat transfer and catalytic reaction in a continuous flow chemical reactor

(Table 4). It can be removed and easily replaced if needed. Advances in 3D printing have allowed for flexible production of parts with specific design requirements for matching fluid properties and flow conditions as well as reduced cost by 10-to-100-fold compared to commercial metal static mixers of similar dimensions (Avril et al., 2017). Electrodeposition (EP) and cold spray (CS) have also been applied in preparation of catalyst-coated static mixers. In addition, some novel techniques such as electron beam additive manufacturing (EBAM) (Wanjara et al., 2019), wire arc additive manufacturing (WAAM) (Derekar, 2018) and laser metal deposition (LMD) (Donadello et al., 2019) have been developed, which are excepted to be applied to this field. Despite their improved heat and mass transfer and reduced pressure drop, there are several challenges facing commercially viable conformation of CSMs, such as deposition quality and repeatability.

Table 4

Some examples of CSMs intensified hydrogenation reactions.

Reaction system	Catalyst	Deposition methods	Reference
Hydrogenation of oleic acid, vinyl acetate, and cinnamaldehyde	Ni, Pt	EP and CS	Avril et al. (2017)
Hydrogenation of vinyl acetate	Ni, Pd	EP and CS	Nguyen et al. (2017)
Reductive amination of aldehydes and ketones-two-step	Ni, Pd	EP and CS	Genet et al. (2018)
Hydrogenation of substituted nitrobenzene	Pd	EP	Gardiner et al. (2018)
Semi-hydrogenation of alkynes	Ni, Pd	CS	Kundraa et al. (2020)

3. Alternative energy sources

3.1. Centrifugal control

It is one of the effective methods for process intensification by applying a controllable external field. In most cases, increasing gasliquid mass transfer rates can significantly improve reaction performance of the reaction process limited by external mass transfer. The high gravity technology ($Gro\beta$ et al., 2019), realized via generating centrifugal force at high-speed rotation, can significantly increase the gas-liquid contact area and gas-liquid mass transfer rate because liquid, within this process, is sheared into tiny droplets, liquid film or ligaments (Yang et al., 2016). Based on this concept, reactors were developed, including rotating packed beds (RPBs) (Hacking et al., 2020), high-shear reactors (Feng et al., 2019) and spinning disc reactors (Martínez et al., 2017), among which RPBs are the most widely studied and applied reactors.

Compared with traditional TBRs, the most significant advantages of RPBs are decrease in time for achieving steady-state operation and feasibility for high viscosity fluids (Neumann et al., 2018). Mixing and mass transfer rates are 1–3 orders of magnitudes higher than those in traditional reactors (Wang et al., 2019). However, because of multi-scale geometric characteristics of RPBs, structural characteristics of packings and flow properties of fluids, it is difficult to obtain the flow phenomenon in reactors and traditional packed bed models are also not suitable for RPBs. Therefore, more and more studies are focus on RPBs via CFD (Ouyang et al., 2019), which makes gas-liquid interaction more understandable and lays the foundation for transitions the technology from lab-scale to industrial scale.

3.1.1. Microwave (MW)

Compared with external heating mode, MW radiation generates effective internal heating by directly coupling MW energy with solvents, reagents or catalysts in reaction, especially when applying MW absorbing materials or receptors, such as silicon carbide (Sun et al., 2019). MW heating has already been applied widely in heterogeneous catalytic reaction (Li et al., 2020) because of increased reaction rates and selectivity, energy saving, improved process control, as well as reduced heat transfer limitations and reduced undesired side reaction (Sturm et al., 2014). Romano et al. (2016) demonstrated high conversion and selectivity by applying a pressurized MW reactor in catalytic hydrogenation of biomass-derived compounds. The combination of MW and microstructure device is promising for process intensification (Yesiloz et al., 2017), although it is still yet limited inconclusive parameters caused by nonuniform thermal distribution (Durka et al., 2010) as well as scaleup issues such as limited penetration depth (Benaskar et al., 2011).

3.1.2. Radio frequency (RF) energy

Compared to conventional heating method, RF heating could improve heat transfer, minimize energy loss, realize a better control in reaction temperature and avoid hot spots caused by directly transferring energy to magnetic catalyst surface (magnetic nanoparticles) (Houlding and Rebrov, 2012). RF heating at intermediate frequencies is promising in some systems because of its greater penetration in comparison to MW heating (Ye et al., 2020). Chatterjee et al. (2015) developed a radio-frequency heating microtrickle bed reactor for catalytic hydrogenation reaction, with which increased total product yield is achieved. For further application of TBR coupled with RF heating, issues including lack of understanding on RF heating, available controllable magnetic nanoparticles and understanding of magnetic actuation still need improved.

3.1.3. Magnetic control

External magnetic field is also considered as a potential method for process intensification and sustainable innovation concepts. Li et al. (2015) demonstrated enhanced catalytic activity when the surface spin configuration of Pd nanoparticles changes from paramagnetic-like to ferromagnetic-like configuration. Magnetic field has also been applied to the fluidized beds. With the development of microfluidics (Yang et al., 2016), controlling the fluid in microfluidic channels by magnetic force has attracted lots of attention because of its enhanced mixing (Boroun and Larachi, 2020a, b) and improved mass transfer (Kumar et al., 2019). However, there are few reports on the enhancement of TBRs by applying a controllable magnetic field.

3.1.4. Ultrasound

Ultrasound, with high frequency and strong penetrating force, could improve the reaction performance of heterogeneous catalytic reaction via created cavitation (Floris et al., 2013) and chemical effects (Maghami et al., 2015). Disselkamp et al. (2005) proved that catalytic activity increased 9 times by applying ultrasound in hydrogenation of cinnamaldehyde. In microreactors, ultrasound could also enhance the generalizability via enhancing mixing (Wang et al., 2013), improving mass transfer (Hübner et al., 2012) and preventing blocking in the microreactor (Hartman et al., 2010). Thus, it is suggested as a promising method for improving gasliquid mass transfer (Yang et al., 2020), with which the total mass transfer coefficient increases by 3–20 times (Dong et al., 2015). However, relatively high energy loss and increased temperature caused by heat dissipation still need to be further addressed.

4. Application of intensified TBRs in petroleum industry

4.1. Petroleum refining

With continuous global oil exploitation, amount of light oils is constantly decreasing and crude oil has gradually developed towards high metals content, high sulfur content and heavy quality. The techniques for upgrading petroleum fractions are one of the most important processes in the refining industry to fulfill requirements of energy saving and environmental protection, as well as to improve use performance of oil. Among the above techniques, hydrotreatment (HDT) shows ability in removing sulfur, metals, nitrogen, asphaltenes and increasing quality and quantity of refined petroleum products (Balasubramanian and Pushpavanam, 2008).

HDT is usually carried out in TBRs, in which HDS, HDN, HDM, HAD, and HDC are often implemented simultaneously. Therefore, development of a detailed, easy-to-operate and highly efficient TBR process model which can be used for design and optimization of the HDT process is practically important for improving fuel quality. Sadighi (2016) demonstrated a two-dimensional (2D) CFD model by using discrete lumping approach to evaluate the yield of a pilot scale vacuum gas oil HDC reactor and validated it by using the reported experimental data in the literature. Novaes et al. (2017) developed a phenomenological model to estimate the kinetic parameters of diesel HDT process, by taking axial dispersion and effectiveness factors for both HDS and HDN reaction into account. Dasgupta and Atta (2020) enhanced HDN process by applying periodic flow modulation in TBRs and developed an isothermal 2D axisymmtric Eulerian model to simulate the intensified HDN process. Gheni et al. (2020) developed a novel dynamics model for evaluating nan-catalyzed HDS process in TBRs and scaled up to a commercial scale.

Nadeina et al. (2017) developed a novel CoMo catalyst for selective hydrotreating of FCC gasoline, which makes it possible to produce a hydrotreated gasoline with sulfur content of less than 10 ppm. Tian et al. (2018) studied the effect of catalyst activity on the energy consumption on a 900000 ton y⁻¹ gasoline-diesel HDT unit by applying the integration of HDT reactor and heat exchanger network and identified optimal matching scheme in terms of both operating cost and capital cost. Saha et al. (2019) developed Ni–Mo/Al₂O₃ and Co–Mo/Al₂O₃ catalysts by cryochemical coprecipitation methods, studied their performance for HDS of dibenzothiophene and obtained a better performance in terms of reaction conversion as well as regeneration. Safari and Vesali-Naseh (2019) designed and optimized HDS of liquefied petroleum gas (LPG) by Aspen Plus software and obtained a cleaner LPG for sulfur reduction to 10 ppm and eliminated all the drawbacks with current HDS of LPG in South Pars Gas Complex.

The average sulfur content ranges from 0.03 wt% to 7.89 wt% in crude oil (Soleimani et al., 2007), whose classification is shown in Fig. 8. Although HDS is the most common sulfur removing method, it is difficult to remove benzothiophene (BT) and dibenzothiophene (DBT) because of high resistance of BT and DBT in hydrogenation process (Nawaf et al., 2015a) and required extreme operating conditions such as high pressures, high temperatures, high volume of hydrogen and more active catalysts. Therefore, alternative deep desulfurization processes have been developed, mainly including adsorption (Blanco-Brieva et al., 2010), extraction (Seeberger and Jess, 2010), oxidation (Jiang et al., 2011) and biodesulfurization,

among which oxidative desulfurization (ODS) is one of the most promising desulfurization methods.

ODS has received a good deal of attention in the recent years because of its advantages, mainly including mild reaction conditions, absence of hydrogen and the ability of removing organosulfur compounds not destroyed in HDS. Nawaf et al. (2015b) developed catalysts of Co_3O_4/γ -Al₂O₃ and MnO_2/γ -Al₂O₃ for ODS of light gas oil containing DBT with air as oxidant, achieved complete oxidation of DBT and obtained high quality fuel for light gas oil. Zhao et al. (2015) developed phthalocyanine molecular sieve catalysts, including Cu₂(PcAN)₂-W-HZSM-5 and Cu₂(PcTN)₂/W-HZSM-5, for ODS of fuel oil and obtained removal efficiencies for DBT of up to 87.32%. The ultrasound-assisted oxidative desulfurization method has been introduced in the desulfurization process, which obtained higher desulfurization efficiency within a shorter reaction time compared with conventional mixing ODS process (Jalali and Sobati, 2017).

4.2. Petrochemical

TBRs have been extensively utilized in oxidation, hydrogenation and hydration in petrochemical, including hydrogenation of alkenes and alkynes, hydrogenation to alcohols, oxidation of ethanol, formic acid and acetic acid and organic pollutants in wastewater.



Fig. 8. Important classification of sulfur-containing compounds in crude oil.

Gorshkova et al. (2012) simulated the hydrogenation process of octene in Ni/Al₂O₃ in TBRs and developed a comprehensive model involving catalytic reaction, mass transfer and heat transfer. Erjavec et al. (2013) enhanced catalytic wet air oxidation of bisphenol A by applying titanate nanotube-based catalysts and achieved total organic carbon removal of 70%. Kasperczyk et al. (2019) removed hydrogen sulphide and volatile organic compounds present in the exhaust air by applying a compact trickle-bed bioreactor and achieved removal efficiencies of up to 97%.

5. Conclusions

As an important type of reactors for gas/liquid/solid catalytic reaction, TBRs are widely applied in petroleum industry, biochemical, fine chemical and pharmaceutical industries. However, TBRs have become increasingly unable to meet current and future industrial production needs because of uneven fluid distribution, non-uniform temperature distribution as well as low mass and heat transfer rate, low yield and temperature runaway caused by hot spots. Furthermore, according to increasingly stringent regulations of acceptable emission concentrations in petroleum industry for reducing the effects of severe weather such as haze, it is necessary to implement techniques capable of fulfilling these requirements. Therefore, process intensification of TBRs becomes increasingly important for efficient industrial production.

Since the 21st century, performance enhancement of TBRs has become one of the important research topics in multiphase reactor engineering. This article reviews recent achievements in process intensification in heterogeneous catalytic reaction in TBRs from five sections, including operating methods and techniques, solvent, catalyst, reactor structure and alternative energy sources. It is obvious that these techniques are very significant for improving mass and heat transfer performance in heterogeneous catalytic reaction. Despite these efforts, several key open questions remain: (1) the feasibility and cost of newly intensified designs, (2) reactor scaling-up, (3) the influence of fluid distribution that varies with operation and design of TBRs, on the mass and heat transfer performance, and (4) the mechanism of alternative energy sources to enhance heterogeneous catalytic reaction. Further studies are required to fulfill the ever-increasing demands along with a sustainable and eco-friendly production techniques.

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