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Advances and Application of Microreactor Technology in Fine Chemistry

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Abstract. In recent years, the global market for fine chemicals has witnessed sustained growth. However, conventional batch reactors employed in fine chemicals synthesis are increasingly confronted with challenges concerning safety, efficiency, and environmental impact, particularly in the production of highly active and hazardous compounds. As an inherently safer process intensification technology, microreactor technology, characterized by its micron-scale channels, high heat and mass transfer efficiency, and continuous flow operation, offers a revolutionary solution for the synthesis of fine chemicals and the transformation of the fine chemical industry. This review aims to summarize recent advances in the application of microreactor technology in key organic reactions, including diazotization, epoxidation, peroxidation, and nitration, and to discuss prospective directions for its future development.

Keywords: Microreactor, Continuous flow, Mass/heat transfer, Fine chemical

1. Introduction

In recent years, the global fine chemicals market has experienced sustained expansion, propelled by societal progress and the emergen of strategic emerging industries. As a core, high-end segment of the chemical industry, the development of fine chemicals is underpinned by the dual drivers of global industrial upgrading and technological revolution. Since the mid-20th century, developed countries have strategically reoriented their chemical industry towards high-value-added, high-technological-barrier fine chemicals, establishing industrial systems centered on specialty chemicals and high-performance materials. Serving as critical foundational materials for strategic emerging industries, fine chemicals underpin innovation in fields such as semiconductors, biomedicine, environmentally friendly additives, and new energy. Their high-value-added characteristics substantially enhance the value density of industrial chains. Furthermore, breakthroughs in green synthesis processes are propelling the chemical industry towards low-carbon and sustainable transformation, making fine chemicals a key indicator of a nation's comprehensive competitiveness in the chemical industry [1].

Nevertheless, significant challenges persist in the manufacturing processes of fine chemicals. Firstly, the synthesis of certain highly reactive products and intermediates involves hazardous reactions including nitration, diazotization, and high-pressure hydrogenation. These reactions are

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typically highly exothermic, with heat flux densities potentially reaching10^3-10^4 W/m^2. The use of conventional batch reactors in such cases presents substantial explosion risks due to slow heat removal [2]. Secondly, these processes frequently employ hazardous chemicals such as strong acids, strong bases, and highly toxic reagents, often under high-temperature and high-pressure conditions, which accelerates equipment corrosion. Furthermore, some reaction by-products exhibit high toxicity or persistent organic pollutant characteristics, posing long-term environmental hazards if not handled properly. Currently, common industrial mitigation strategies include reducing reactant concentrations, limiting reactor dimensions, and lowering reaction temperatures to prevent thermal runaway [3]. However, as national regulations increasingly classify common fine chemical processes as priority-controlled hazardous chemical operations, the development of the fine chemical industry is significantly constrained. This constitutes a key limiting factor for the industry's green transition, presenting severe challenges to corporate production safety and environmental protection.

Microreactors represent miniature chemical reaction systems fabricated based on micro-processing technology. Their defining characteristic is micron-scale reaction channels (typically 10-1000 micrometers), constructed via techniques such as laser etching or photolithography on substrates including silicon, glass, or metals to form three-dimensional flow structures. Primary system components comprise microchannel units, auxiliary modules, and fluid control systems [4]. As a typical representative of inherently safer process intensification technology, microreactors offer a high surface area-to-volume ratio, enabling effective management of strongly exothermic reactions. The continuous flow operation and minimal reaction volumes inherent to microreactors provide intrinsic safety advantages, rendering them suitable for various high-risk reactions, such as rapid exothermic processes and nitration, thereby demonstrating considerable adaptability and flexibility [5,6].

Substantial research has confirmed the extensive application and significant impact of microreactor technology in fine chemistry, where it is catalyzing transformative changes [7]. This review comprehensively examines fundamental research and recent advances in microreactors applied to reactions such as diazotization, epoxidation, peroxidation, and nitration, addressing safety and efficiency issues associated with these hazardous processes and providing reference for further research.

2. Diazotization and azo-coupling reaction

Diazotization refers to a reaction in which aromatic primary amines react with sodium nitrite under low temperatures conditions and in the presence of excess strong acid to generate diazonium salts. This reaction is widely utilized in the synthesis of azo pigments, dyes, sulfonamide drugs, and antibiotics. Nevertheless, diazonium salts are unstable and explosive. Furthermore, unreacted reagents and residual by-products during the reaction may present significant hazards to ecosystems and human health, which severely constrains the industrial application of diazotization [8,9]. Microreactor technology, characterized by high heat and mass transfer efficiency as well as effective micromixing, provides a safer and more efficient synthetic pathway for diazotization reactions [10]. Integrating diazotization and coupling reactions within a microreactor system can significantly improve reaction yield and selectivity, thereby reducing energy consumption and shortening reaction time [11]. For homogeneous systems, due to the excellent fluidity of reactants, implementation at the microchannel scale is relatively straightforward. Presently, various commercial azo dyes have been successfully synthesized in microreactors. However, when employing raw materials or pigment products exist in a suspended or particulate form, reactor channel clogging invariably results in

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failure [12]. Wang et al. [13] utilized a microreactor system for the continuous synthesis of two major azo pigments, namely Pigment Red 146 and Pigment Yellow 14. Their research found that for the synthesis of Pigment Red 146, the design of an improved micro-sieve dispersion mixer could solve the clogging issues encountered in conventional microreactors, resulting in a high conversion rate of 96.99%. In their another study, they employed a three-stream micromixing process to precisely control the reaction pH of Pigment Yellow 14 synthesis, enabling the production of pigments with high purity (>95%), small particle size, and narrow particle size distribution [14].

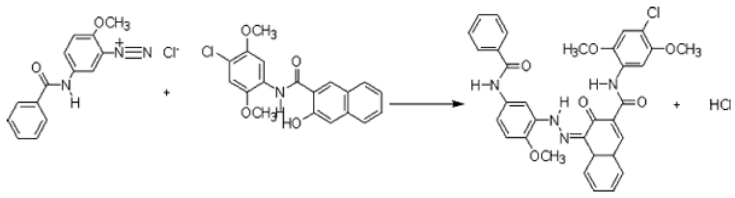

Figure 1. Schematic of the synthesis pathway of Pigment Red 146 [13]

3. Epoxidation reaction

Epoxidation denotes a category of chemical reactions in which alkenes are converted into epoxides, typically involving gas-liquid or liquid-liquid heterogeneous reactions. For such reactions, the mass transfer rate at the phase interface crucially determines the reaction kinetics. Epoxidation is an important reaction for synthesizing various fine chemicals. For instance, styrene oxide, the epoxidation product of styrene, serves as a key intermediate in the synthesis of pharmaceuticals and other fine chemicals. Pennemann et al. [15] employed 2,2,2-trifluoroacetophenone to catalyze the epoxidation of styrene with an oxidant (e.g., hydrogen peroxide) under alkaline conditions. This reaction in confronted with challenges including strong exothermicity (>100 kJ/mol) and heterogeneous catalysis, thus necessitating intensified mixing to eliminate diffusion limitations, increase the interfacial contact area, remove reaction heat, and prevent the decomposition of hydrogen peroxide under alkaline conditions [16]. To address these issues, researchers developed a continuous-flow microreactor system, which effectively controlled the reaction heat and prevented significant hydrogen peroxide decomposition, achieving both high conversion (>95%) and high selectivity (>95%) for styrene oxide [17]. They also proposed a reaction kinetic model to guide the optimization of the reaction process.

Due to the inherent advantageous properties of microreactors, they can maintain high reaction efficiency even at relatively lower temperatures (e.g., 40-60 ℃) [18]. Furthermore, microreactor systems enable more precise control of reaction conditions (such as H_2O_2 dosing rate), reducing side reactions. In contrast, conventional batch reactors exhibit slower reaction rates under identical conditions and are prone to product degradation caused by local overheating [19].

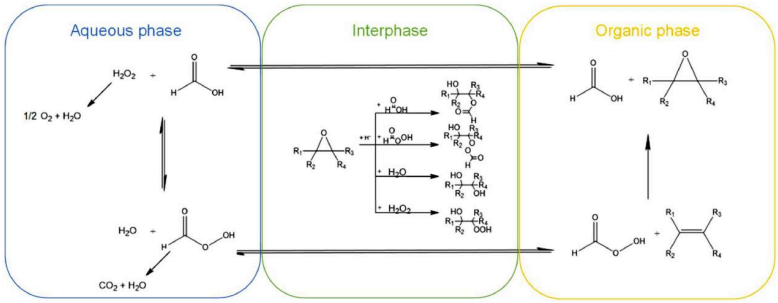

Figure 2. The epoxidation of soybean oil [19]

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4. Peroxidation reaction

Peroxidation reactions constitute a class of chemical transformations that introduce a peroxy group (-O-O-) to form organic peroxides. These reactions are typically conducted under acidic conditions. However, a crucial issue associated with the generated peroxides is their poor stability, rendering them susceptible to decomposition or explosion due to temperature fluctuations, mechanical friction, or deviations in feedstock ratios. Given these serious challenges, a novel peroxidation process utilizing microreactor technology has been developed. Xia et al. [20] investigated the synthesis of tert-amyl hydroperoxide (TMHP) and they found that when a semi-batch reactor was employed, the substantial heat release and inefficient heat transfer necessitated a slow reagent addition rate. Furthermore, excessive heat accumulation during the process could lead to a runaway reaction. In contrast, the adoption of a microreactor system has been demonstrated to realize the continuous-flow synthesis of TMHP, significantly improving both process safety and efficiency, attributed to its superior heat transfer performance. Moreover, the microreactor process achieved a product yield comparable to that of conventional batch reactors, while requiring a lower sodium hydroxide equivalent [21].

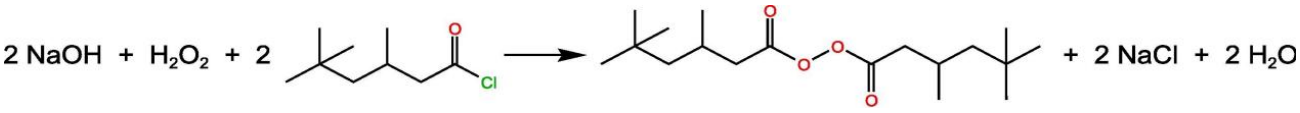

Figure 3. Chemical reaction equation of TMHP synthesis [21]

In another study, Li et al. [22] examined the intensified synthesis mechanism and reaction kinetics of diisobutyryl peroxide in a microreactor. Their findings indicated that the enhanced mass transfer characteristics of the microreactor significantly accelerated the peroxidation rate and suppressed by-product formation. Additionally,, the system's excellent thermal stability was found to restrict the maximum temperature rise to less than 5 ℃. Through the optimization of reaction parameters (e.g., temperature, reactant concentration, and residence time), high product selectivity could be maintained even at low temperatures, effectively avoiding the local hotspots and decomposition risks prevalent in conventional batch reactions. Kinetic studies revealed that the peroxidation in the microreactor followed a pseudo-first-order reaction model, with a key finding that the apparent activation energy was significantly lower than that in conventional reactors, further validating the remarkable advantages of microreactor technology in improving reaction efficiency and safety from both thermodynamic and kinetic perspectives.

5. Nitrification reaction

Nitrification refers to the chemical process of introducing a nitro group ($-NO_2$) into an organic molecule. This reaction is characterized by intense heat release, concentrated exothermicity, and a high reaction rate, which can readily lead to explosion if not properly controlled [23]. Additionally, these reactions are generally accompanied by side reactions including the formation of polynitro compounds and oxidation processes. Therefore, efficient heat removal and precise control of the nitration temperature are critical factors in determining process safety.

The primary limitation of obtaining accurate kinetic data for nitrification lies in their rapid, highly exothermic nature and tendency to undergo side reactions, thereby rendering measurement by conventional techniques challenging. Nevertheless, previous studies have established the advantages of microreactors in investigating the kinetics of heterogeneous nitration reactions [23,24]. In a study

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aiming to determine the nitration kinetics of 4-methyl-2-nitrotoluene (4-MNT) under conditions of continuous flow and homogeneous nitration in a microreactor, Song et al. successfully acquired the relevant kinetic data. The results of the study show that the microreactor system, owing to its superior mass transfer performance and precise temperature control (±1℃), enhanced the selectivity for 2,4-dinitrotoluene to 92% while reducing by-product formation by 40-60%. It was verified that the nitration reaction conformed to a pseudo-first-order kinetic model. Interestingly, the reaction rate constant in the microreactor was 2-3 times higher than that in a conventional batch reactor, a phenomenon attributed the highly efficient heat and mass transfer at the microscale. When the nitric acid concentration exceeded 90%, the microreactor effectively avoided side reactions caused by local overheating, highlighting its unique advantage in enabling the safe implementation of highly hazardous nitration reactions.

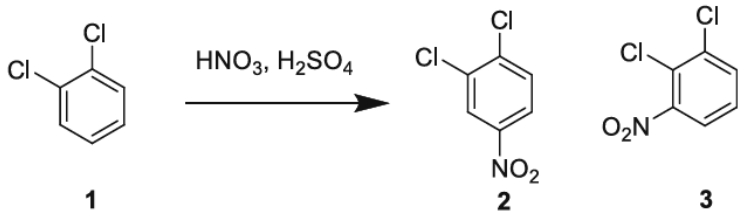

Figure 4. Nitration reaction of o-dichlorobenzene [24]

6. Other reactions

In addition to the aforementioned reactions, microreactors have been demonstrated to yield substantial performance enhancements in other reactions such as cycloaddition, alkylation, and sulfonation. In the case of cycloaddition reactions, for example, the synthesis of propylene carbonate from $CO_2$and propylene oxide exhibited markedly improved reaction efficiency through intensified mass transfer in a microreactor, achieving a product yield of 99.7% [25]. Similarly, Okamoto et al. [26] reported the realization of electrochemical [3+2] cycloaddition of phenols and olefins in a laminar flow microreactor, with the electrolyte concentration minimized to 0.001 $mol \cdot L^{-1}$. Regarding alkylation reactions, Yang et al. [27] investigated the synthesis of long-chain alkylated naphthalene from naphthalene and α-olefins, using chloroaluminate ionic liquids as catalysts. They showed that a microreactor system achieved a yield exceeding 99% within 60 seconds at 30 ℃. Furthermore, Wang et al. [28] conducted a series of experiments on the alkylation of isobutane/1-butene catalyzed by [OMIm][OTf]/H_2SO_4 in a microreactor. This work successfully demonstrated that an alkylate with a research octane number (RON) of 99.5 could be obtained at 10.0 ℃ with a reaction time of 81 seconds. With respect to sulfonation reactions, Geng et al. [29] applied SO_3 as the sulfonating agent for the synthesis of dodecylbenzene sulfonic acid (DBSA) in a microreactor. By optimizing the reaction conditions, they achieved high reaction rates and selectivity, with a product purity exceeding 97%. In another investigation focusing on gas-liquid sulfonation, Xu et al. [30] found that a yield of 89.8% for fatty acid methyl ester sulfonate was achieved in a microreactor under an annular flow pattern, with a molar ratio of 1.2 and a reaction temperature of 80℃.

Collective evidence from these studies indicates that the application of microreactors under appropriate conditions can substantially improve reaction yield and efficiency. Furthermore, numerous studies have quantified the substantial impact of operational parameters—such as temperature, pH, reagent concentration, and flow rate—on reaction yield and selectivity. Thus,

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further optimization of these parameters represents a viable strategy for enhancing both the efficiency and safety of these processes.

7. Conclusions

This review has systematically examined the advancements and current state of microreactor technology for various fine chemical syntheses. The findings reveal that the demonstrated high efficiency, environmental friendliness, and improved safety fundamentally stem from the inherent advantages of microreactors, specifically, their superior mass and heat transfer characteristics and precise control over reaction conditions. These attributes have led to promising application potential across multiple fields. For instance, in the chemical industry, microreactors are widely utilized in organic synthesis, inorganic synthesis, and the preparation of polymer material. In bioengineering, they are extensively applied in areas such as cell culture, genetic engineering, and protein crystallization studies. In the pharmaceutical sector, microreactors are integrated throughout the entire workflow, encompassing drug discovery, synthesis process optimization, and analytical testing. Recent advances have witnessed domestic researchers fabricate high-throughput borosilicate glass microreactors, which have significantly enhanced equipment stability and reaction efficiency. Concurrently, the research conducted by Professor Marc Pera-Titus's team at Cardiff University has focused on exploration of novel catalysts and reaction systems, where new strategies such as surface-active catalytic particles have been developed to overcome mass transfer limitations, thereby providing efficient solutions for various reactions [31]. Looking ahead, it is anticipated that the continuous evolution of Internet of Things (IoT) technology will drive microreactors toward higher level of intelligence and automation. A key implication of this trend is that integrated remote control and real-time monitoring systems will substantially improve data accuracy and operational efficiency. Furthermore, the multifunctional andintegrated platform characteristics of microreactors are expected to expanded, integrating process including multi-step synthesis, separation, purification, and online analysis. This study has demonstrated that such technological advancement will not only strengthen the process adaptability of microreactors but also facilitate the transition of fine chemicals, biomedicine, and related industries towards greener and smarter production paradigms.

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