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Emerging trends in the selective production of value-added aromatics

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1 Introduction

Aromatics (mainly benzene, toluene, and xylenes, BTX) are essential intermediates in the chemical industry. They are employed in the synthesis of a broad spectrum of chemicals and materials, e.g., various polymers, plastics, resins, solvents, dyes, pharmaceuticals [1]. The manufacturing pathway of BTX mainly includes petroleum reforming of naphtha, coal-to-aromatics, methanol-to-aromatics [2]. On one hand, the production capacity of paraxylene (PX) has increased to more than 70 million tons per year during the past decade, with an average annual growth rate above 6%. China became a major player in the BTX industry, producing more than 45% of global PX in 2022. On the other hand, the global market of BTX is facing pressure from over-supply and relatively low prices due to the downward economics and epidemic [3]. The average operating rate of aromatic complex plants dropped below 75% due to the declining global market.

In the era of the energy revolution from traditional systems based on fossil fuels to a new energy landscape based on renewable resources, refining and chemical enterprises are attempting to re-modulate their product pipelines, transitioning from a fuel producer towards a fuel and/or chemical supplier [4]. Besides BTX, the exploitation of heavy aromatics has drawn much attention, especially for aromatic compounds with boiling points of >190 °C. Versatile aromatics such as durene, naphthalene and anthracene are receiving great attention in the chemical industry because they can be further converted into chemicals as monomers in the downstream polymer industry for the production of high-performance polymers as well as liquid organic hydrogen carriers for H_2 storage and transport.

Herein, we attempt to discuss the new trends in the catalytic transformation of value-added aromatics and derivatives from a combination of academic and industry perspectives. We will list some emerging reactions in the selective conversion of monocyclic and bicyclic aromatics into chemicals with increasing interest in downstream processes.

2 Emerging value-added aromatic derivatives

2.1 Toluene derivatives

As one of the aromatic products with very high global production, toluene has very broad applications in the chemical industry for the preparation of a variety of key commodity chemicals. Beyond the conventional downstream derivatives, we would like to highlight the selective transformation of toluene into several value-added derivatives (as described in Figure 1).

One of the toluene's major applications is used as the precursor for the preparation of para-xylene, which is further transformed into terephthalic acid as a monomer of poly-

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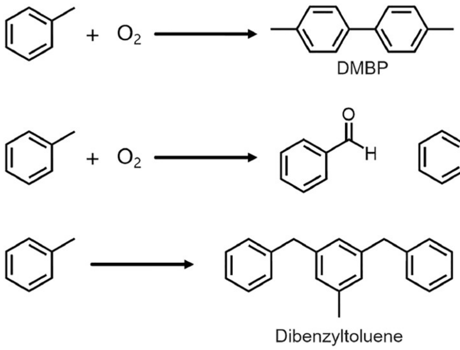

Figure 1 Conversion of toluene into value-added derivatives.

ethylene terephthalate (PET). As an analogue of para-xylene, 4,4′-dimethylbiphenyl (DMBP) can be transformed into derivatives through selective oxidation reactions (Table 1), and the products can be used as plasticizers and monomers of polyesters. In principle, DMBP can be synthesized from toluene through an oxidative coupling reaction based on homogeneous Pd catalysts (Figure S1) [5]. Expensive ligands are required to achieve substantial yields, but the low chemoselectivity to the desired product and the difficulty in catalyst and ligand recycling preclude the large-scale application of this direct synthesis approach. Other routes for the preparation of DMBP from toluene involve multiple steps of alkylation, dehydrogenation, and isomerization reactions, resulting in high energy consumption and low process efficiency. Recently, it has been reported that confining Pd active sites in the 12-ring channels of Beta zeolite can achieve high regioselectivity in direct aerobic oxidative coupling of toluene to DMBP thanks to the shape-selectivity properties of Beta zeolite structure, enabling an atom-economic synthesis of DMBP [6]. This concept can also be extended to the preparation of supported metal catalysts for coupling of toluene and biomass-derived furan compounds, which can be further converted into DMBP through Diels–Alder and dehydration reactions [7]. These new synthesis routes open the possibility of low-cost and sustainable preparation of DMBP, but the design of highly efficient and robust catalysts is the key to realizing practical application [6,8–18].

Through oxidative reaction, toluene can be converted to oxygenates such as benzyl alcohol and benzyl aldehyde, which are also value-added chemicals [19]. Currently, the selective oxidation of toluene is mainly achieved in the liquid phase via a radical mechanism, which brings difficulty in controlling the product distributions. It has been reported that by using a ligand-modified iron oxide catalyst, it is possible to achieve aerobic oxidation of toluene to benzyl aldehyde with >99% selectivity at ~35% toluene conversion at 180 °C [20]. If this process can be scaled up, benzyl alcohol can also be obtained by selective hydrogenation of benzyl aldehyde, opening a new possibility of production of high-purity benzyl alcohol.

Dibenzyltoluene (DBT) is considered a very promising liquid organic hydrogen carrier (LOHC) for H_2 storage and transportation applications based on the reversible transformation between dibenzyltoluene and fully hydrogenated dibenzyltoluene (18H-DBT) [21]. Traditionally, DBT is widely used as a heat transfer fluid and can be produced through the alkylation reaction between toluene and benzyl chloride in the presence of an $AlCl_3$ catalyst (Table 1). A considerable amount of chloride impurity remains in the DBT, which may cause serious problems in the LOHC system because of the formation of Cl-containing compounds (e.g., HCl) in the H_2 storage procedure through the DBT hydrogenation reaction, resulting in catalyst deactivation and reactor corrosion. Therefore, it is of great necessity to develop new synthesis routes for the preparation of Cl-free DBT or low-cost methods for the removal of Cl-containing impurities in the DBT prepared by conventional method. For instance, Cl-free DBT can be prepared by the alkylation reaction between toluene and benzyl alcohol on solid acid catalysts [22,23]. To improve the yield of DBT in this process, the morphology of the zeolite catalysts should be modulated to facilitate the diffusion of the reactants and products (Figure S2).

2.2 Durene derivatives

The polymethylated aromatic durene (1,2,4,5-tetramethylbenzene, TeMB) has drawn much attention in recent years because its oxidized form (pyromellitic dianhydride, PMDA) is an essential raw material for fabricating heat-resistant plastics polyimide (PI), which is widely applied in the aerospace, electronics, and national defense systems (see molecule structures in Figure 2). Currently, China is the world's largest manufacturer and consumer of durene, consuming more than 95% of the industrial production of PI with a steady growth rate over the last decade.

A substantial amount of durene is isolated in high purity from catalytic reforming streams through the combination of distillation and crystallization methods [24]. For instance, a typical composition of reforming C10 aromatics contains >11 wt.% of durene. Meanwhile, the synthetic routes to durene based on the methylation or disproportionation of pseudocumene (trimethylbenzene, TMB) and isomerization of TeMBs have been developed, which substantially reduce the production cost, as pseudocumene can be easily recovered in high purity by fractional distillation from petroleum sources [11,25].

Similar to the classic alkylation of toluene, the methylation of pseudocumene with methanol can be catalyzed by metal-impregnated HZSM-5 (Table 1). Specifically, methanol was activated by the Brønsted acidic site to form a methoxy group, and the TMB molecule was subsequently attacked by the methoxy cation to form protonated TeMB [26]. The main

Table 1 Summary of literature review on synthesis method of DMBP, DBT, durene, 2,6-DMN and EN

Main reactions	Catalysts	Reaction conditions	Conversion (%)	Selectivity (%)	Ref.
Oxidative coupling of toluene into DMBP	$Pd(OAc)_2$ H-Beta	110 °C, 1.6 MPa, O_2 AcOH as the additive	38	65	[6]
Alkylation of toluene and benzyl chloride into DBT	$FeCl_3$	Atmospheric pressure and temperature	Yield: 78.0%–87.9%		[8]
Methylation of 1,2,4-TMB and methanol into durene	HZSM-5	300–500 °C, 0–0.8 MPa, methanol/1,2,4-TMB=1–3 mol mol^{-1}	30–50	40–55	[9]
Disproportionation of 1,2,4-TMB into durene	SSZ-33 zeolite, NU-87 zeolite or aluminium chlorohydroxyl complex	200–400 °C, atmospheric pressure, methanol/1,2,4-TMB=1–3 mol mol^{-1}	10–65	41–80	[10–12]
Alkenylation of *o*-xylene and butadiene into 5-(*o*-tolyl)-2-pentene	K/Al_2O_3	139–143 °C	22–50	22–54	[13]
Cyclization and dehydrogenation of 5-(*o*-tolyl)-2-pentene into 1,5-DMN	Mordenite zeolite and Pd/C	250–300 °C, atmospheric pressure,	100	>90	[14]
Isomerization of 1,5-DMN into 2,6-DMN	Y zeolite	250 °C	39–91	/	[15]
Isopropylation of naphthalene into 2,6-DIPN	Mordenite zeolite	250 °C, 0.8 MPa	80	65	[16]
Methylation of 2-methylnaphthalene and methanol into 2,6-DMN	MCM-22 zeolite	350 °C, methanol/methylnaphthalene=1, WHSV=0.5 h^{-1}	58	14	[17]
Ethylation of naphthalene and ethanol in to EN	Y, MCM-44 or SAPO-5	250 °C, WHSV=0.5 h^{-1}, naphthalene: ethanol=1:5 mol mol^{-1}	31–73	44–76	[18]

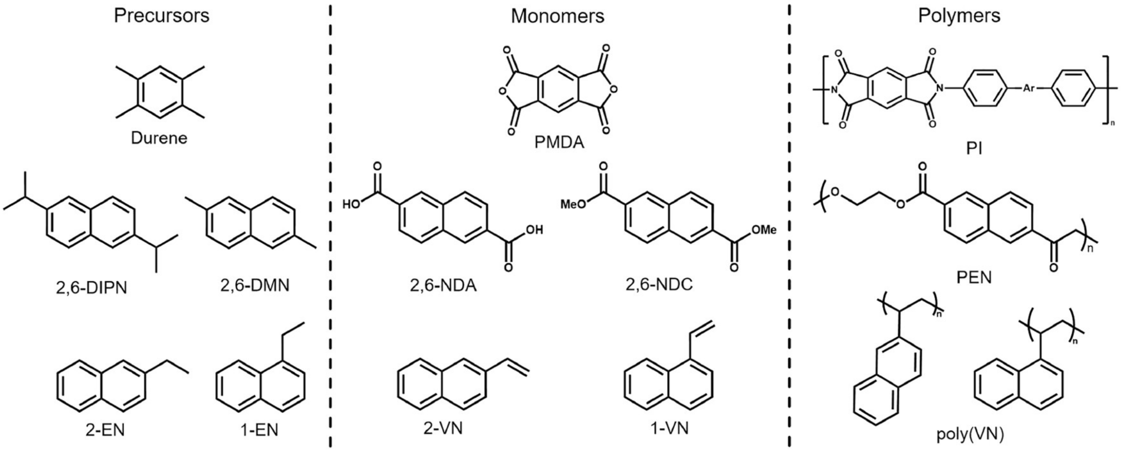

Figure 2 Conversion of aromatics into value-added monomers for downstream polymer industry.

challenge in this process is to suppress the side reaction of methanol dehydration into olefins, due to the high reaction temperatures (350–380 °C). Alternatively, to eliminate the usage of methanol as the alkylation agent, syngas is introduced with pseudocumene into the reaction system in the presence of a bifunctional catalyst, where the CO hydrogenation and TMB methylation occur tandemly on different active sites [27]. The coupling reaction allows for higher alkylation efficiency and product selectivity, on account of the inhibited side reactions including excessive alkylation, tetramethylbenzene isomerization, and the water–gas shift reaction. The spatial distribution and the number of active sites should be carefully modulated to maximize the synergy of the bifunctional catalyst composite in the tandem reaction process.

Disproportionation of pseudocumene can be conducted in a pressurized fixed-bed reactor, generating TeMB and xylene isomers through a bimolecular reaction pathway over large-pore zeolites (e.g., Y and Beta zeolite) with three-dimensional pore structures (Table 1) [10,11]. The high durene selectivity can be attributed to the small pore dimension of the 12-ring pores in Y and Beta zeolite that hinders the free exit of bulky 1,2,3,5-TeMB isomer [28]. It should be noted that mordenite (MOR zeolite) with the one-dimensional 12-

ring channel shows dominated transition state shape selectivity, promoting the formation toward *p*-xylene rather than its *ortho*/*meta* isomers. From a materials perspective, tuning the nanoscale morphology of zeolite catalysts is a promising approach to modify the product distributions in this catalytic process.

2.3 Naphthalene derivatives

Naphthalene is the simplest polycyclic aromatic compound and its derivatives (methyl-naphthalene and other alkylated-naphthalene) serve as precursors in the field of drug synthesis and material science. In particular, dimethylnaphthalene (DMN) is one of the most demanding aromatic hydrocarbons as its oxidised form (2,6-naphthalenedicarboxylic acid, 2,6-NDA) and its dimethyl ester (dimethyl-2,6-naphthalenedicarboxylic ester, 2,6-NDC) have been long sought after as the precursors for high-performance polyester polyethylene naphthalate, featuring higher modulus, outstanding heat resistance and improved mechanical stability [29]. Although dimethylnaphthalene is relatively abundant in fossil resources (up to 15 wt.% in coal tar), it exists as a mixture of isomers that can hardly be isolated at affordable cost. Thus, the production of 2,6-DMN cannot rely solely on the separation from coal tar or light catalytic oil (LCO) on account of its lack of adequate availability and high cost [30]. Many alternative chemical routes to produce 2,6-DMN have been developed over the past decades. *o*-Xylene-butadiene condensation route was first commercialized by BP Amoco in the 1990s, with over 60 million lb per year capacity. It involves multi-step catalytic reactions promoted by catalysts with different functions, including alkenylation, cyclization, dehydrogenation and isomerization (Table 1). From a practical point of view, such processes require sophisticated operations and do not match the green chemistry principles.

Alkylation routes beginning with naphthalenic compound show economic advantage because reaction steps could be omitted for naphthalene ring construction. The isopropylation of naphthalene (Table 1) over zeolites to fabricate 2,6-diisopropylnaphthalene (2,6-DIPN) is reported with satisfying selectivity to the desired product because the bulky alkylating agent introduces more steric effect as the second propylene attaches to isopropylnaphthalene [16,31]. However, as the substituted alkyl group becomes larger, the following oxidation to 2,6-NDA becomes more difficult with less theoretical yield. In this sense, the methylation of refinery streams containing naphthalenic compounds is considered a promising route to lowering the cost of 2,6-DMN production, especially when the feedstock contains a large proportion of methyl naphthalenes.

The methylation of naphthalenic compounds can be achieved over the MCM-22 zeolite catalysts (Table 1), in which the acid sites on the external of the MCM-22 zeolite account for the alkylation reaction [32]. By optimizing the properties of acid sites in the MCM-22 catalyst, the selectivity to 2,6-DMN can be improved by suppressing the side reactions caused by the coke deposition and undesired isomerization reactions. Although the selectivity to 2,6-DMN is primarily controlled by thermodynamics in the alkylation reaction and is relatively lower than that in the 2,6-DIPN route, the final yield to 2,6-DMN can be compensated by incorporating the isomerization and transalkylation reactions as complementary processes for reforming DMN isomers or tri-methylated by-products into 2,6-DMN [33]. In addition, this methylation route also takes advantage of the low-cost feedstock, such as light cycle oil, coke gas oil, coal tar and catalytic reforming residue oil, which are at the edge of excess capacity in the current petrochemical industry. Challenges remain on the catalyst iteration in terms of reaction stability and tolerance of polar impurities (especially when using the feedstocks made with mixed aromatics) and the systematic optimization of the elementary processes.

Ethylnaphthalene (EN) can be transformed into vinylnaphthalenes (VNs) via the dehydrogenation, generating value-added precursors in the pharmaceutical and dye industry. Meanwhile, the polymerization of vinylnaphthalene is of great importance, because the naphthalene rings will change the optical and mechanical properties of the polymers [34]. Due to the low abundance of ethylnaphthalene in the feedstock from fossil resources, the synthesis of vinylnaphthalene is mainly acquired via the hydrogenation of acetyl naphthalene, followed by the dehydration in the presence of the polymerization inhibitor. However, the acetyl naphthalene is prepared by conventional Friedel–Crafts catalysts and large-scale production cannot be guaranteed due to the harsh reaction conditions. As an environmentally friendly route, the ethylation of naphthalene over zeolite catalysts could be reconsidered (Table 1). Large pore zeolites with high acidity and surface area, including Y, MMW, Beta, and MOR, are candidate catalysts for efficient conversion of naphthalene with satisfying selectivity towards mono-ethylatednaphthalenes [18]. To suppress the formation of naphthalene products with multiple ethylation reaction sites, the reaction conditions and the microstructures of the zeolite catalysts should be carefully optimized.

Replacing ethanol with long-chain alcohol in the alkylation of naphthalene will lead to alkylated naphthalene to empower lubricants to deliver durability and optimum performance in many operating environments. Furthermore, the alkyl naphthalene sulfonate compounds are classified as biodegradable surfactants with low toxicity, which have broad applications as industrial and alkaline cleaners, photographic chemicals, and printing. The alkylation of naphthalene with long-chain alcohols requires the use of solid acid catalysts with large pore sizes to avoid diffusion limitations.

2.4 Other emerging aromatics

Besides the abovementioned compounds, some other aromatics and derivatives with high added values in the chemical industry deserve attention from researchers in academia, whose market capacity could be smaller than the abovementioned commodity chemicals but their manufacturing processes call for innovations in catalysis science and technologies. For instance, the polyurethane industry requires the use of methylenedianiline (MDA) as a crucial intermediate in polyurethane manufacturing. By replacing the HCl with solid acid catalysts, it is feasible to greatly decrease the capital investments in reactor construction in MDA production units. A recent study shows that hierarchical LTL-type zeolite exhibits an unprecedentedly high selectivity to 4,4′-MDA and high stability for the condensation reaction between aniline and formaldehyde, making it a compelling solid acid catalyst for substituting the conventional HCl in the MDA production unit [35].

Another research trend in processing aromatic feedstocks is developing catalytic routes for utilization of biomass-derived aromatics. Due to the high structural diversity of biomass-derived aromatics and the complicated chemical reactivities, the selective conversion of biomass platform molecules into commodity chemicals is of great challenge and importance to achieve a carbon-neutral society [36]. Some interesting processes include the production of xylenes from the addition reaction of ethylene and 2,5-dimethylfuran and the selective conversion of lignin-derived aromatics into value-added chemicals [37].

Considering the rapidly increasing production of green H_2 from renewable electricity and the supply of CO_2 from carbon capture process, the integration of CO_2 reduction into the aromatic manufacturing processes is an attractive route for sustainable utilization of renewable hydrogen and carbon resources [38]. Some potential reactions in this direction include the direct alkylation of benzene and toluene with CO_2 and H_2, resulting in the formation of toluene and xylenes, respectively, without the addition of methanol. The development of bifunctional catalysts made of active sites for CO_2 hydrogenation and aromatic alkylation reactions is vital to achieve the tandem conversion [26].

3 Summary and outlook

In this Perspective, we have briefly discussed the emerging trends in converting commodity aromatics into value-added chemicals and materials. Limited by the article length, we only cover some typical transformation routes with relatively large production capacities and broad applications. It should be noted that, due to the very high diversity of monomers in the polymer industry, there are many more interesting and industrially relevant transformation routes not covered in this article. In particular, due to the excess supply of BTX in recent years, developing low-cost and flexible processes to modulate the isomer ratios in the BTX mixture is critical for maintaining the profit level of the major chemical companies. Some promising directions include the coupling of reforming process with the utilization of excess light alkane and CO/CO_2 resources [39,40]. Though most of the processes involved in BTX production have been intensively studied for decades, there are still some spaces in catalyst discovery and process optimization, especially with the aid of artificial intelligence [41,42]. We believe that an intimate collaboration between industry and academia is critical to figuring out promising research topics in this field [43]. With the industry–academia synergy, innovations in catalyst design and process/reactor engineering will enable the development of sustainable processes for the utilization of aromatic resources. A typical research paradigm can be practiced with the translation of the novel catalysts developed by academic researchers into practical reactions dealing with aromatic feedstocks. The technical issues encountered in the testing of the new catalysts under industrially relevant conditions can further inspire the optimization and upgrading of the catalysts.

Beyond the direct translation of the knowledge and experiences accumulated in fundamental studies, the researchers in industry can also participate in the frontier research activities in academia by providing practical insights into the design of the reaction routes and the scaling up of catalyst preparation and reaction process. It is reflected in the literature that many interesting reactions dealing with the conversion of aromatics (e.g., direct oxidation of benzene to phenol with O_2, direct amination of benzene to aniline with NH_3, direct carboxylation of benzene to benzoic acid with CO_2) have been intensively studied for decades [44,45]. Close collaborations between academia and industry will facilitate their adoption in practical processes, or at least the knowledge translation into other related fields.

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Conflict of interest The authors declare no conflict of interest.

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