

Donor–Acceptor Type Conjugated Microporous Polymer as a Metal-Free Photocatalyst for Visible-Light-Driven Aerobic Oxidative Coupling of Amines

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Abstract

Developing cheap, highly efficient, metal-free heterogeneous photocatalysts remain a great challenge in photoredox reactions. Herein, we utilize a typical Suzuki coupling of low-cost triphenylamine derivative and 9,10-dibromoanthracene to synthesis a donor–acceptor type conjugated microporous polymer (denoted as PAA-CMP). As expected, heterogeneous PAA-CMP exhibits excellent photocatalytic performance, good functional group tolerance and satisfying recyclability in metal-free aerobic oxidative coupling of amines to imines driven by visible light, which is due to its absolute energy level positions and good physicochemical stability. More excitingly, PAA-CMP can enable the gram-scale air-oxidized photocatalytic conversion under natural sunlight irradiation, yielding the desired imine product with an isolated yield of 65% for 48 h. The current work provides a great application prospect for CMPs in low-cost and large-scale organic industrial production in the future.

Graphic Abstract



The prepared donor-acceptor (D-A) type conjugated microporous polymer (PAA-CMP) as a metal-free and heterogeneous photocatalyst enable the gram-scale air-oxidized photocatalytic conversion of benzylamine into N-benzylidenebenzylamine under natural sunlight irradiation, yielding the desired imine product with an isolated yield of 65%.

Keywords Conjugated microporous polymer · Donor-Acceptor · Heterogeneous photocatalysis · Oxidative coupling

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1 Introduction

Visible-light-induced photoredox catalysis has emerged as one of the most attractive approaches to achieve the transformation of organic compounds into high-value-added intermediates due to its high chemoselectivity and the operation under mild reaction conditions (e.g. room temperature and ambient pressure) [1–3]. Up to now, numerous photocatalysts involving inorganic semiconductors (TiO₂ [4, 5], In₂O₃ [6], Au–Pd alloy [7], etc.), transition metals-based complexes (Ru [8], Pd [9], Pt [10], etc.) and organic dyes (eosin Y [11], Flavin [12], rose bengal [13], etc.) have been successfully employed to photocatalytic organic transformations, such as coupling of amines, oxidation of sulfides, dehydrogenation of *N*-heterocycles, hydroxylation of arylboronic acids, oxidation of aldehydes, etc. Regardless of their high efficiency, there are some inherent weaknesses including high cost, toxicity and instability, thereby limiting their application in large-scale industrial production. Therefore, the development of various cheap, metal-free, stable and heterogeneous photoredox systems is an effective strategy to overcome these limitations.

Conjugated microporous polymers (CMPs) as a class of potential heterogeneous photocatalytic platform have attracted growing attention in photoredox catalysis (e.g. coupling of amines [14], oxidation of sulfides [15], dehalogenation of haloketones [16], stille-type coupling [17], Aza-Henry reaction [18], etc.) due to their extended π -conjugated skeletons, wide visible-light absorption, high stability, non-toxicity and repeatability. It is generally believed that photocatalytic performance of CMPs is likely determined by their energy band structure (namely the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO)) [19]. Research shows that a precise combination of electron donor-acceptor (D-A) moieties in CMPs can not only optimize the energetic band level but also enhance separation of photogenerated charge carriers, thus improving their photocatalytic performance [20–22]. Recently, some D-A type carbazole-based CMPs synthesized via a known oxidative polymerization have displayed great potential in several photoredox reactions such as degradation of mustard-gas [23], Aza-Henry reaction [24], C-3 functionalization of indoles [25], and 1,2-formylarylation of N-arylacrylamides [26]. Because of strong electron donating ability of carbazole units, these carbazole-based CMPs show significantly more positive HOMO position and more negative LUMO position compared to the other analogues, indicating the presence of their strong redox abilities. Nevertheless, these chosen monomers usually suffer from tedious synthesis procedures and high costs. In this regard, the direct polymerization of electron-donor and electron-acceptor monomers is a more intriguing and cost-effective approach for fabricating a series of new CMPs with D-A structure. For instance, Zhang's group has utilized benzothiadiazole as an electron acceptor to prepare a series of D-A type CMPs via metal-assisted cross-coupling reactions [27].

Herein, we employ a low-cost electron-donor triphenylamine derivative and an electron-acceptor 9,10-dibromoanthracene as raw materials to prepare a D-A type CMP (PAA-CMP) via a typical Suziki-coupling reaction. As expected, PAA-CMP is found to be an excellent metal-free heterogeneous photocatalyst for aerobic oxidative coupling of primary amines into various imine products with high photocatalytic activity, good functional group tolerance and satisfying recyclability. More interestingly, PAA-CMP has the ability to achieve the gram-scale photocatalytic oxidation by using benzylamine as a substrate and air as an oxidant under natural sunlight irradiation, implying that PAA-CMP may possess a great potential for the application of CMPs in large-scale organic industrial production in the future.

2 Experimental Section

2.1 Synthesis of PAA-CMP

A 250 mL three-necked flask equipped with a spherical condenser was charged with tris-[4-(4,4,5,5-tetramethyl-[1,3,2] dioxaborolan-2-yl)-phenyl]-amine ((PhBpin)₃A, 0.33 mmol) and 9,10-dibromoanthracene (Br₂A, 0.5 mmol), and then degassed N,N-Dimethylformamide (DMF, 50 mL) and K₂CO₃ solution (2 M, 10 mL) were added under N₂ atmosphere. The mixture was stirred and heated up to 80 °C, and then *tetrakis*(triphenylphosphine)palladium (Pd(PPh₃)₄, 0.017 mmol) was added under N₂ atmosphere. The resulting mixture was stirred and heated up to 150 °C for 72 h. After cooling to room temperature, the formed precipitate was filtered and washed with water, acetone and tetrahydrofuran, respectively. Further, the collected solid was purified via soxhlet extraction with tetrahydrofuran solution and finally dried at 100 °C in vacuum oven to give a yellow-green powder (89% yield, 0.33 g, containing Pd of 0.63 wt%).

2.2 Photocatalytic Oxidation Coupling of Amines

A quartz tube equipped with a cold finger condenser was charge with photocatalyst PAA-CMP (15 mg), substrate amine (0.5 mmol), *n*-dodecane (0.5 mmol) as the internal standard and acetonitrile (10 mL). The reaction mixture was conducted with ultrasonic dispersion for 10 min, and then was stirred and irradiated by white LED under O_2 atmosphere at 25 °C for 16 h. After the reaction was accomplished, photocatalyst was recycled by centrifugation, and the supernatant liquid was analyzed by GC to confirm yield of target product through the calibration curves. In scale-up reaction for oxidative coupling of benzylamine, the imine product was obtained by filtration, concentration and silica gel chromatography with ethyl acetate.



Fig. 1 Synthesis of PAA-CMP

3 Results and Discussion

3.1 Characterization

As shown in Fig. 1, PAA-CMP was synthesized via a typical Suzuki coupling reaction between $(PhBpin)_3A$ as a donor unit and Br_2A as an acceptor unit. Scanning electron

microscopy (SEM) images show that PAA-CMP is composed of spherical microparticles of different sizes (Fig. S1). Successful coupling reaction was also confirmed by FT-IR and solid-state¹³C CP/MAS NMR. Figure 2a shows that the C–Br peak (577 cm⁻¹) in Br₂A and B-O peak (1359 cm⁻¹) as well as CH₃ group (2978 cm⁻¹) of boronic pinacol ester in (PhBpin)₃A are almost absent from the spectrum



Fig.2 a FT-IR spectra of (PhBpin)₃A, Br₂A and PAA-CMP; **b** Solid state ¹³C CP/MAS NMR spectrum of PAA-CMP; **c** Absorption isotherms of PAA-CMP under N₂ at 77 K; **d** Pore size distributions of PAA-CMP

of PAA-CMP [28-30], testifying to effective Suzuki coupling. Solid-state¹³C CP/MAS NMR spectrum of PAA-CMP reveals peak at 146 ppm arising from carbon atoms in C-N bonds and peak at 135 ppm arising from carbon atoms (e and f) in anthracene ring (Fig. 2b). In addition, signals at 129 and 125 ppm are attributed to carbon atoms in benzene ring and anthracene ring. Adsorption-desorption measurement of PAA-CMP under N2 at 77 K exhibits Brunauer–Emmett–Teller (BET) surface areas of 506 m² g⁻¹ with the total pore volume of $0.59 \text{ cm}^3 \text{ g}^{-1}$ (Fig. 2c). Based on nonlocal density functional theory (NLDFT), pore size distribution of PAA-CMP is calculated to be around 0.8, 1.1 and 1.6 nm, demonstrating microporosity (Fig. 2d). Thermal analysis demonstrates that PAA-CMP retains about a 98% weight up to 530 °C, confirming its high thermal stability (Fig. S2).

UV-vis diffuse reflectance spectrum (DRS) of PAA-CMP, which well inherits the feature of acceptor Br_2A , exhibits a sufficient absorption band ranging from 300–500 nm (Fig. 3a). This permits PAA-CMP to be availably excited under visible-light irradiation. Under excitation at 270 nm, both acceptor Br_2A and polymer PAA-CMP have almost same maximum emission peak around 505 nm, and emission intensity of PAA-CMP is evidently weaker than acceptor Br₂A (Fig. 3b). At the same time, UV-vis DRS and emission spectra of PAA-CMP are significantly red-shifted than those of donor (PhBpin)₃A. The red shift as well as the decreased emission intensity possibly signifies the effective intramolecular energy transfer and electron transfer [20, 24, 31, 32], which is mainly derived from the donor-accepter structure of PAA-CMP. To elucidate the electronic properties of PAA-CMP, cyclic voltammetry (CV) measurement was implemented to reveal its energy band structure. As shown in Fig. 3d, the lowest unoccupied molecular orbital (LUMO) position of PAA-CMP is determined to be -1.75 V vs. Ag/AgCl. With the bandgap value of PAA-CMP calculated to be 2.67 eV from Kubelka–Munk function (Fig. 3c), the highest occupied molecular orbital (HOMO) position of PAA-CMP is then determined to be 0.92 V vs. Ag/AgCl (inset in Fig. 3d). Because the LUMO potential of PAA-CMP is much more negative than the reduction potential of O_2/O_2^{-} (-0.48 V vs. Ag/AgCl) [33, 34], it is theoretically feasible for photocatalytic activation of O2 to generate active species superoxide radical anion (O_2^{-}) over PAA-CMP, thereby conducting oxidative coupling of amines.



Fig.3 a UV–Vis DRS spectra of $(PhBpin)_3A$, Br_2A and PAA-CMP; **b** Steady-state photoluminescence emission spectra of $(PhBpin)_3A$, Br_2A and PAA-CMP (excitation at 270 nm); **c** Band gap of PAA-

CMP according to the Kubelka–Munk theory. **d** Cyclic voltammogram of PAA-CMP at a scan rate of 100 mV/s; Cut in the image is energy diagram of the HOMO and LUMO positions of PAA-CMP

3.2 Photocatalytic Performance

To investigate the photocatalytic performance of PAA-CMP, we first conducted the aerobic oxidative coupling of benzylamine into N-benzylidenebenzylamine under white LED lamp irradiation, and the results are listed in Table 1. The blank experiments showed that the oxidation reaction did not proceed in the absence of light, oxygen or catalyst (Table 1, Entries 1–3), signifying that these factors including light, oxygen and catalyst are essential for oxidative coupling of benzylamine. Under white LED irradiation and O2 atmosphere, PAA-CMP could promote the transformation of benzylamine into N-benzylidenebenzylamine with an 86% yield (Table 1, Entry 4). To investigate which are active species in this reaction, some scavengers including potassium iodide (KI), 1,4-diazabicyclo[2.2.2]octane (DABCO) [35], benzoquinone and isopropanol were added to verify the presence of the hole (h^+), singlet oxygen (${}^{1}O_{2}$), O_{2}^{--} , hydroxyl radical (·OH), respectively. It was shown that KI, DABCO and benzoquinone could effectively quench the reaction, while isopropanol did not play inhibiting effect (Table 1, Entries 5-8), suggesting that crucial species involved in oxidative coupling of amines are composed of h⁺, ¹O₂ and O₂⁻⁻ except ·OH. Importantly, compared to commercial photocatalysts such as anatase TiO₂, BiVO₄ and g-C₃N₄ (Table 1, Entries 9-11), the designed D-A type PAA-CMP showed the highest photocatalytic activity for oxidative coupling of benzylamine. Particularly, anatase TiO₂ also could promote conversion of benzylamine into N-benzylidenebenzylamine with medium yield, which was attributed to slight ultraviolet light around 285 nm of white LED.

Next, we explored the substrate scope of oxidative coupling of primary amines, and the results are listed in Table 2. Benzylamines bearing electron-donating groups (e.g. methoxy and methyl) were transformed into the corresponding imines with higher yields than benzylamines bearing electron-withdrawing groups (e.g. bromo and trifluoromethyl) under the same reaction conditions (Table 2, Entries 2–5). This is possibly because that electron-donating group enhances electronic density of the substrate, which is easily oxidized by defective-electron hole of PAA-CMP. Para-substituted benzylamine showed higher reactive activity than ortho-substituted benzylamine because of steric hindrance (Table 2, Entries 2 and 6). Moreover, picolylamine and 2-thiophenemethylamine, which are regarded as representative of heterocyclic amines, could be converted into the corresponding imines with 91% and 85% yields, respectively (Table 2, Entries 7 and 8).

Further, PAA-CMP which was simply recovered by centrifugal separation was again added to benzylamine solution to investigate its recyclability. As shown in Fig. 4a, PAA-CMP could be used at least six cycles without significant loss of catalytic activity, exhibiting high recyclability. Most of FT-IR spectra of PAA-CMP before and after six cycles are consistent (Fig. S4A). However, in-plane bending vibration at 1390 cm⁻¹ of residual CH₃ group and out-plane bending vibration at 882 cm⁻¹ of aromatic C-H in fresh PAA-CMP are disappeared after six

Entry	$\begin{array}{c c} & \text{photocatalyst, O}_2 \\ \hline & White LED \end{array} \end{array} \longrightarrow \begin{array}{c} & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & $					
	Photocatalyst	O ₂	hν	Scavengers ^a	Inhibited Species	Yield (%) ^b
1	PAA-CMP	+	_	_	_	n.d
2	PAA-CMP	_	+	-	_	1
3	_	+	+	_	_	n.d
4	PAA-CMP	+	+	_	_	86
5	PAA-CMP	+	+	KI	h^+	40
6	PAA-CMP	+	+	DABCO	$^{1}O_{2}$	43
7	PAA-CMP	+	+	benzoquinone	02	19
8	PAA-CMP	+	+	isopropanol	·OH	86
9	TiO ₂	+	+	-	_	57
10	$BiVO_4$	+	+	-	_	66
11	$g-C_3N_4$	+	+	_	_	25

 Table 1
 Photocatalytic aerobic oxidative coupling of benzylamine into N-benzylidenebenzylamine

Benzylamine (0.5 mmol), photocatalyst (15 mg), *n*-dodecane (internal standard, 0.5 mmol), O_2 atmosphere, white LED, acetonitrile (10 mL), 25 °C and 16 h

^aScavengers (0.5 mmol)

^bDetermined by GC with *n*-dodecane as the internal standard

 Table 2
 Substrate Scope for oxidative coupling of primary amines into imine products



Primary amine (0.5 mmol), photocatalyst (15 mg), *n*-dodecane (internal standard, 0.5 mmol), O_2 atmosphere, white LED, acetonitrile (10 mL), 25 °C and 16 h

^aDetermined by GC with *n*-dodecane as the internal standard





Fig. 4 a Recyclability of PAA-CMP in oxidative coupling of benzylamine into *N*-benzylidenebenzylamine with *n*-dodecane as the internal standard; **b** Scale-up reaction under nature sunlight and air atmosphere. Reaction conditions: benzylamine (18.7 mmol, 2 g),

PAA-CMP (100 mg), air atmosphere, acetonitrile (100 mL), nature sunlight irradiation for 48 h in Guangzhou, China (07/13/2020–07/14/2020, atmospheric temperature (28–35 °C))

cycles (Fig. S4B), suggesting that residual groups (e.g. boronic pinacol ester) in PAA-CMP easily fall off during the photocatalysis reaction. To explore the applicability of PAA-CMP for further industrial manufacture, a gram scale reaction for the oxidative coupling of benzylamine

into *N*-benzylidenebenzylamine was performed under nature sunlight irradiation and air atmosphere. As shown in Fig. 4b, after proceeding for 48 h, an isolated yield of 65% for *N*-benzylidenebenzylamine was obtained. These results indicate that PAA-CMP possess great application prospects for low-lost and large-scale organic transformation.

3.3 Mechanistic Considerations

To shed light on the mechanism of photocatalytic oxidative coupling of amines, some control experiments were conducted. N, N, N', N'-tetramethylphenylenediamine (TMPD), a well-known rich-electron indicator, is widely employed to evaluate the ability of photocatalyst with respect to mediating electron transfer from electron-donating substrates to oxygen [36]. As shown in Fig. 5A, after white LED irradiation for 0.5 h, PAA-CMP could promote the formation of strong absorption peaks at 523, 562 and 614 nm assigned to cationic radical species of TMPD, but the solution color turned from without color to purple color rather than the reported blue color [37], which is possibly attributed to the emission spectrum of white LED containing slight ultraviolet light (Fig. S1). This result indicates that PAA-CMP can mediate electron transfer from the amine substrate to O_2 , thereby resulting in active species O_2^{-} . Next, hydrogen peroxide (H_2O_2) , one of the postulated intermediates, was investigated by using Horseradish peroxidase (POD)-catalyzed oxidation of N, N-diethyl-1,4-phenylenediammonium sulphate (DPD) [38]. In the presence of PAA-CMP, two strong absorption peaks at 512 and 553 nm along with obvious color change from light red to dark red were formed (Fig. 5b), probably indicating the presence of H_2O_2 in catalytic system of PAA-CMP. Further, ${}^{1}O_2$, one of the vital reactive oxygen species derived from energy transfer in photoredox reactions, was detected by electron spin resonance (ESR) test with 2,2,6,6-tetramethylpiperidine (TEMP) as the trapping agent. Figure 5c showed that PAA-CMP promoted the generation of a strong 1:1:1 triplet signal, which was assigned to 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) radical derived from reaction between TEMP and ${}^{1}O_{2}$ [39].

In view of the above experimental results and reported literatures [25, 40–42], one plausible mechanism involving both O_2^{--} and 1O_2 was proposed for PAA-CMP catalyzed oxidative coupling of benzylamine. As shown in Fig. 5D, photocatalyst PAA-CMP was excited to produce the excited state (PAA-CMP^{*}) under visible-light irradiation. Then, molecular oxygen (O₂) adsorbed on surface of PAA-CMP was activated by PAA-CMP^{*} to reactive oxygen species O_2^{--} and 1O_2 via electron transfer and energy transfer, respectively, as well as resulting in the formation of cationic radical of PAA-CMP (PAA-CMP⁺⁻), which was also regarded as the hole. Next, benzylamine substrate was



Fig. 5 UV–vis absorption spectra and photograph of the cationic radical species of TMPD \mathbf{a} and DPD \mathbf{b} generated by PAA-CMP in the presence of visible light and oxygen; \mathbf{c} ESR spectra of the samples

after mixing TEMP solution with PAA-CMP and O_2 under visible light. **d** Proposed mechanism of PAA-CMP photocatalyzed oxidative coupling of benzylamine to the imine

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oxidized by positive charge of PAA-CMP⁺ to generate the cationic radical of benzylamine (**a**), which reacted with O_2^{--} or 1O_2 to form the imine intermediate (**b**) and H_2O_2 . Particularly, the in situ formed H_2O_2 could oxidize the free benzylamine to afford the imine intermediate (**b**). Finally, the imine intermediate (**b**) reacted with a free benzylamine to produce the intermediate (**c**), which further gone through elimination of ammonia under hole-assisted to afford the imine product.

4 Conclusion

In summary, we designed and synthesized a class of D-A type CMPs, which was considered as a low-cost, metal-free, heterogeneous photocatalyst in the oxidative coupling of amines into imines. Thanks to its excellent optoelectronic properties and good chemical stability, PAA-CMP with the D-A structure exhibits remarkable photocatalytic activity, good tolerance and satisfying recyclability. Especially, a gram-scale preparation of the imine product was achieved with a high yield of 65% under natural-sunlight irradiation by using benzylamine as a feedstock and air as an oxidant, thus opening the prospect for the large-scale industrial applications of CMPs. Further studies are underway to optimize synthesis approach of D-A type CMPs via metal-free catalysis.

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Compliance with ethical standards

Conflicts of interest There are no conflicts to declare.

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