

Article

Crystalline Carbon Nitride Embedded with Pt Nanoparticles for Boosting Photothermal Degradation of Toluene

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Abstract

Degradation of volatile organic compounds (VOCs) by environmentally friendly methods remains a challenging issue. Photothermal catalysis, as an emerging green catalytic technology, merges the benefits of both thermal catalysis and photocatalysis, presenting itself as a viable strategy for VOC degradation. However, achieving higher catalytic performance by reasonably designing the synthetic route of catalyst carriers remains difficult. In this study, crystalline carbon nitride material, poly(triazine imide) (PTI), was prepared using a unique molten salt synthesis method and employed as a support for Pt to construct an exceptional photothermal catalyst. In a continuous-flow system under Xe lamp irradiation with external temperature control, toluene was efficiently degraded at a high rate of nearly 100% under low Pt content (0.31 wt%) and a relatively low operational temperature condition (143 °C). As a carrier of noble metals, PTI material exhibited a larger specific surface area and fewer structural defects, resulting in more efficient toluene conversion and mineralization. The joint action of photocatalysis and thermocatalysis synergistically facilitated the efficient generation of active species and accelerated charge transfer, thereby significantly boosting toluene catalytic oxidation. These findings provide valuable guidance for designing and optimizing photothermal catalysts for the removal of VOCs.

Keywords: photothermal catalysis; poly(triazine imide); molten salt synthesis; toluene degradation



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1. Introduction

The considerable amount of emissions produced by volatile organic compounds (VOCs) presents a critical hazard to human well-being and the ecological environment [1]. Consequently, exploring efficient and stable technologies of VOC removal has become a significant challenge. In general, the degradation of VOCs can be achieved by photocatalytic (PC) or thermocatalytic (TC) reactions. However, the single operation of PC or TC has some limits. PC often exhibits relatively low reaction efficiency and inadequate oxidation capacity, while TC generally requires high energy consumption and may experience catalyst deactivation, which constrains the potential application scenarios of catalysts [2,3]. Photothermal catalysis (PTC), integrating the attributes of PC and TC, demonstrates enhanced efficiency in catalytic performance [4]. In PTC reactions, thermal energy can effectively facilitate the movement and isolation of photogenerated charges, while the light-induced thermal effect accelerates TC reaction kinetics [5,6]. Moreover, reactive oxygen species generated during

photocatalysis play a pivotal role in driving the complete oxidation of pollutants [7,8]. Therefore, PTC is considered an effective approach in tackling low degradation efficiency and high external energy requirements [9].

Currently, two categories of PTC materials are employed for the elimination of VOCs: catalysts modified by noble metals and catalysts based on metal oxides [10]. Compared with catalysts based on metal oxides, noble metal-modified catalysts have attracted extensive attention due to their superior catalytic performance, enhanced selectivity, and greater stability [11–13], such as Pt/TiO₂ [14], Pt/ γ -Al₂O₃ [15], and Pd-Ag@CeO₂ [16]. Optimizing noble metal-supported catalysts heavily relies on the inherent characteristics of carrier materials, which significantly impact electron transport efficiency and dispersion of active sites [17–21]. Recently, polymerized carbon nitride (PCN) has emerged as a superior catalyst support, recognized for its environmental nontoxicity, exceptional thermal stability, facile tunability and customization, as well as its diverse surface chemistry [22–24]. PCN is a polymer material connected by the bridge amino linkage of the heptazine structure unit with a π -conjugated system via sp² hybridization [25]. The –NH₂, –NH groups on the surface of PCN facilitate the dispersion and stabilization of noble metal nanoparticles through the formation of a strong coordination interaction [26]. PCN could be recognized as a distinguished catalyst in catalytic oxidation reactions [27]. However, owing to the inherently sluggish mass transport in a solid-phase system, the polycondensation reaction during traditional thermal polymerization is often incomplete, leading to many unnecessary structural defects becoming the recombination center of photogenerated carriers, seriously limiting its catalytic efficiency [28,29]. Fortunately, molten salt synthesis provides a better mass transfer rate and reaction environment for more complete polycondensation reactions, thus expanding the π -conjugated system [30,31]. Recent studies have demonstrated that molten-salt-assisted synthesis can promote the formation of more ordered frameworks and tunable microstructures, which effectively regulate the crystallinity, morphology, and surface properties of PCN materials, improving their catalytic performance [32–34]. Compared with the amorphous PCN, the crystalline carbon nitride prepared by the molten salt synthesis method has the advantages of higher crystallinity, more regular morphology, and a larger specific surface area. Its fundamental framework is additionally condensed into a triazine ring structural unit, forming the poly(triazine imide) (PTI) structure [35]. Due to its highly ordered framework and improved structural stability, PTI is expected to exhibit superior catalytic performance compared to conventional amorphous g-C₃N₄-based materials. Although Pt/g-C₃N₄ catalysts have been widely reported for oxidation of VOC [27], the application of crystalline PTI-based catalysts in photothermal VOC degradation remains rarely explored.

In this paper, binary alkali metal chloride LiCl/KCl ($T_m = 352\text{ }^\circ\text{C}$, 45:55 wt%) was selected as the medium for the molten salt synthesis. A series of PTIs with different grain sizes were successfully synthesized through manipulation of the precursor to the molten salt mass ratio, which was used as the carrier for anchoring Pt nanoparticles. Compared with PCN, a superior toluene conversion efficiency was demonstrated by PTIs with higher crystallinity during PTC degradation under continuous-flow conditions. Based on the characterization of PTI crystals, the optimal amount of molten salt medium for the synthesis of PTI with better PTC performance was determined. Furthermore, an investigation into the mechanism underlying this activity enhancement was conducted.

2. Results and Discussion

2.1. Structure and Composition of the Catalysts

The microscopic morphologies of Pt-PTI_x and Pt-PCN samples were determined by SEM and TEM. Figures S2 and S3 display the SEM and TEM images of Pt-PCN samples,

which exhibit a typical bulk accumulation morphology [27]. All Pt-PTI_x samples showed similar hexagonal prisms in Figures 1a–d and S4 [28], but their crystal packing arrangements and particle dimensions varied. As shown in Figure 1a, when the amount of molten salt was 1:1, PTI₁ crystals exhibited extensive disordered aggregation and poor dispersion. This phenomenon occurs because the precursor cannot be completely dissolved in an inadequate molten salt environment, and the inadequate amount of molten salt fails to provide sufficient template space for crystal formation and growth. This has a negative effect on the uniform dispersion of reaction points [36]. As depicted in Figure 1b–d, the increase in molten salt dosage is observed to alleviate the aggregation of PTI crystals, resulting in improved crystal dispersion and a gradual increase in the size of hexagonal prism crystals. The increased molten salt content facilitates a more favorable melting environment and template space for crystal growth, thereby enhancing the dissolution of precursors in the molten salt environment and providing sufficient space for PTI crystal growth. The specific surface area of Pt-PTI₁, Pt-PTI₃, Pt-PTI₅, and Pt-PTI₁₀ in Figure S5a shows an initial rise followed by a subsequent decline, with corresponding values of 39, 54, 28 and 19 m²/g, respectively. Among them, Pt-PTI₃ possesses the highest specific surface area, which could be ascribed to its improved dispersion and relatively smaller apparent particle size. Therefore, adding an appropriate amount of molten salt can affect crystal size and dispersion [37]. In addition, PTI exhibits a markedly higher specific surface area compared to the conventional Pt-PCN (only 9 m²/g), implying greater exposure of active sites. As a porous material, the Pt-PTI₃ sample has the largest pore volume and pore size (Table S2, Figure S5), which can facilitate the adsorption and degradation of reactants in favor of catalytic reaction. The TEM and HRTEM images in Figure 1e,f exhibit a crystal spacing of 0.72 nm for PTI₃, which can be attributed to the (100) crystal plane relative to the in-plane repeating unit, indicating its good crystallinity. In addition, lattice fringes with a spacing of 0.224 nm are observed, corresponding to the Pt (111) plane, confirming the successful deposition of crystalline Pt species. The Pt nanoparticles are estimated to be on the order of ~3 nm in size and are well dispersed on the PTI₃ surface. HAADF-STEM analysis, along with EDX elemental mapping (Figure 1g), demonstrates a homogeneous arrangement of N, C, Cl and Pt elements within the Pt-PTI₃. The relatively small particle size and uniform dispersion of Pt, together with the extremely low Pt loading (0.31 wt%), suggest minimal aggregation, which is favorable for the PTC reaction [38].

The powder XRD technique was utilized to investigate the crystal structures of the Pt-PCN and Pt-PTI_x samples. The crystal diffraction structures viewed in Figure 2a for all Pt-PTI_x show remarkable similarity, which can be attributed to the characteristic diffraction pattern of PTI [31,39]. The peak of diffraction at 12.0° can be attributed to the in-plane periodic arrangement of triazine units, corresponding to the (100) crystal plane with an interplanar spacing of around 0.72 nm. This value is in good agreement with the lattice spacing observed in the HRTEM images, further confirming the ordered in-plane structure of the PTI. Meanwhile, the strongest diffraction peak observed from 26.4° corresponds to the (002) crystal plane with a crystal plane distance of 0.32 nm, representing the stacking of conjugated aromatic rings [40]. In the Pt-PCN sample, the periodic arrangement of heptazine basic structural units and the stacking of graphene layered structures correspond to the two typical diffraction peaks at 13.0° and 27.5°, respectively [41]. The lack of Pt diffraction peaks in the XRD patterns of all samples may be explained by the limited quantity, comparatively tiny particle size, and homogeneous distribution of Pt nanoparticles [27,42]. The diffraction peaks of Pt-PTI_x exhibit a more pronounced sharpness compared to that of Pt-PCN, indicating the significantly enhanced crystallinity of the PTI sample. This improvement in crystallinity is expected to alleviate structural defects of PTI and promote the effective transfer of photogenerated charge.

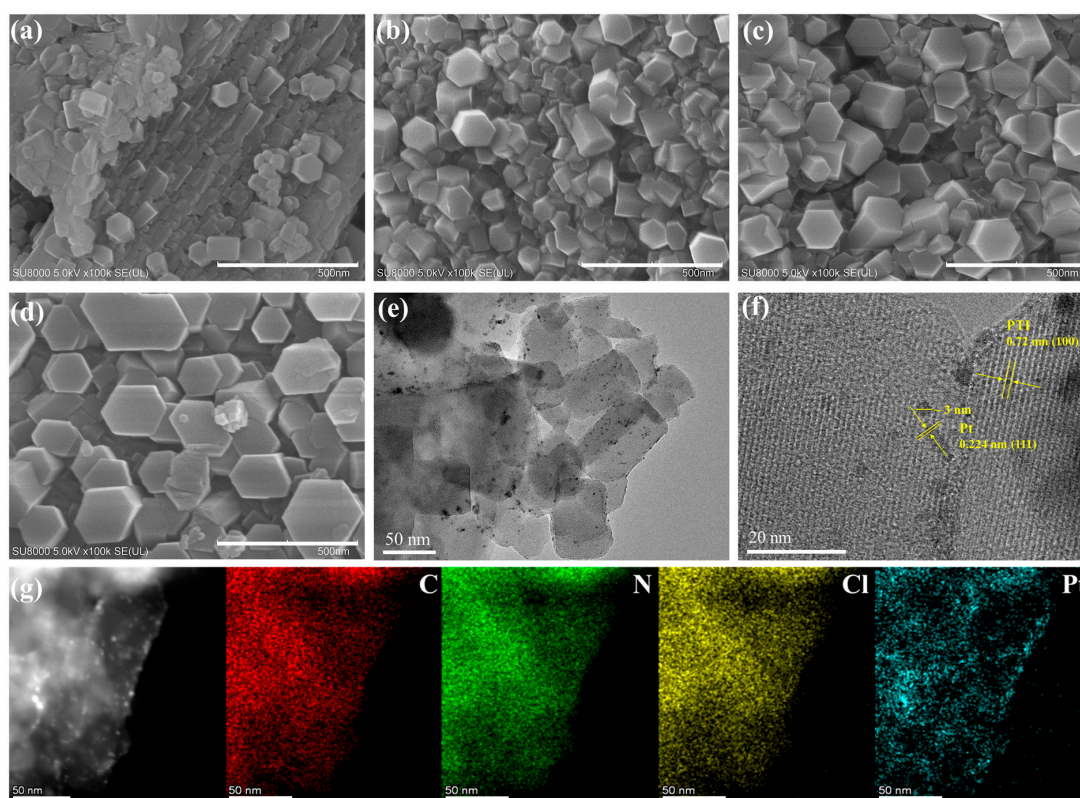


Figure 1. SEM image of (a) Pt-PTI₁, (b) Pt-PTI₃, (c) Pt-PTI₅, and (d) Pt-PTI₁₀ (scale bars: 500 nm); (e,f) TEM image of Pt-PTI₃; and (g) HAADF-STEM and EDX mapping image of Pt-PTI₃ sample (scale bars: 50 nm).

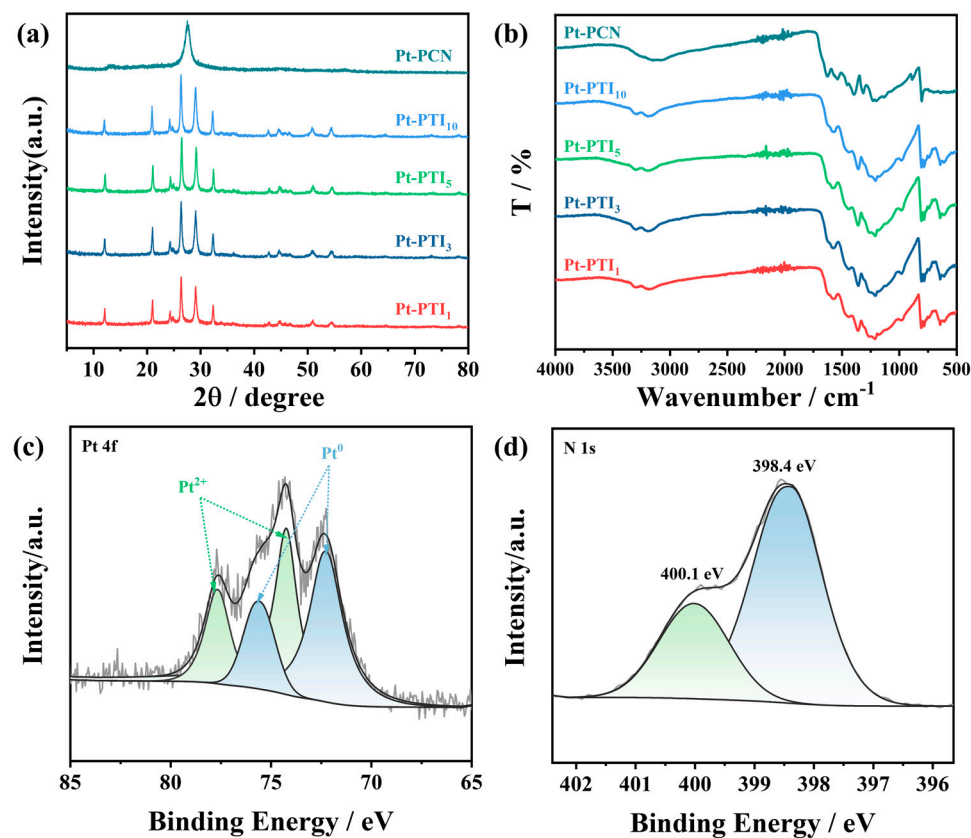


Figure 2. Cont.

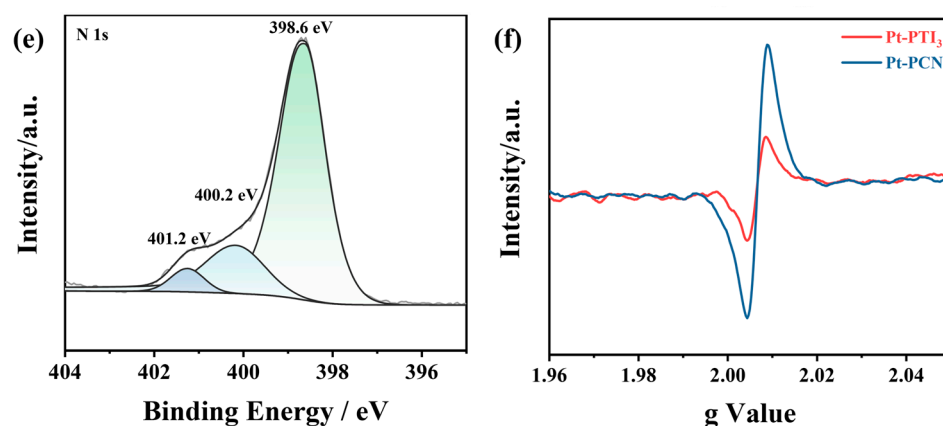


Figure 2. (a) XRD patterns; (b) FT-IR spectrum of Pt-PTI_x and Pt-PCN; XPS spectrum of (c) Pt 4f; (d) N 1s of the Pt-PTI₃; (e) N 1s of Pt-PCN; and (f) EPR spectra of Pt-PTI₃ and Pt-PCN.

To investigate the chemical constituents and surface properties of the sample, we conducted FT-IR, solid-state ^{13}C NMR, Raman spectroscopy, and XPS analysis. In Figure 2b, the FT-IR spectrum reveals that the prepared Pt-PTI_x samples exhibit nearly identical functional groups. The vibrations at 3200 cm^{-1} , $1200\text{--}1600\text{ cm}^{-1}$, and 800 cm^{-1} originate from the stretching vibrations of bridging amino groups and the characteristic vibrational modes of triazine rings, including the out-of-plane bending vibration of the triazine units [43]. The FT-IR spectra of Pt-PCN display similarities to the typical g- C_3N_4 pattern [44]. The triazine-based framework of PTI₃ is further supported by the solid-state ^{13}C NMR spectrum (Figure S6). The resonance signals at approximately 162 and 158 ppm are attributed to the carbon atoms within the triazine rings, while the splitting of these peaks arises from differences in the local chemical environment caused by proton coordination. In addition, the signal located at 168 ppm is assigned to the carbon atoms of triazine units without proton coordination [45,46]. These results indicate that the fundamental structural units of the PTI₃ material are composed of triazine rings rather than heptazine rings. Meanwhile, the chemical states of Pt were analyzed by XPS. The high-resolution Pt 4f spectrum (Figure 2c) displays two sets of peaks, which can be ascribed to the coexistence of both metallic (Pt^0) and oxidized (Pt^{2+}) platinum states due to spin–orbit coupling of Pt 4f electrons. The presence of these species is consistent with previous reports [27,47]. Based on the peak fitting results, the proportion of metallic Pt^0 is calculated to be approximately 76.8%, indicating that Pt mainly exists in a metallic state on the catalyst surface. Pt species have been proven to be active sites in PTC reactions, which can promote electron transfer, oxygen adsorption, and the generation of reactive oxygen species in toluene degradation [38,48,49]. In the PTI sample, the N 1s spectrum (Figure 2d) was deconvoluted into two peaks at 398.4 eV and 400.1 eV, with the former assigned to sp^2 hybridization nitrogen ($\text{C}=\text{N}=\text{C}$) in the triazine ring and the second one assigned to the sp^3 hybridization of the bridge N atom ($\text{C}=\text{NH}=\text{C}$) within its structure [50]. In contrast, the Pt-PCN sample exhibited the deconvolution of the N 1s spectrum (Figure 2e), which revealed three distinct nitrogen species. The peak at 398.6 eV was characteristic of the sp^2 -hybridized N atom presented within the heptazine ring ($\text{C}=\text{N}=\text{C}$). Additionally, another peak appeared at 400.2 eV, which originated from the N atom forming the bridging units in the heptazine ring ($\text{N}=\text{C}_3$). The peak at 398.6 eV is characteristic of the two-coordinated sp^2 nitrogen (N_2C) within the triazine ring, while the peak at 400.2 eV originates from the three-coordinated nitrogen (N_3C) that links the triazine rings. Lastly, a third peak emerged at 401.2 eV originating from an incompletely polymerized amino group located on the surface of the sample ($-\text{NH}_x$) [44,51]. The XPS results confirm that PTI has a more complete polycondensation structure and its π -conjugated system is more extensive, which reduces unnecessary structural defects and provides a

more efficient channel for charge migration. For the LiCl/KCl eutectic mixture used in the synthesis process, the PTI samples were scanned by a high-resolution spectrum of Li and Cl elements (Figure S7b,c). The results show that the presence of Li and Cl species is associated with the PTI framework, which is attributed to the residual molten-salt components remaining after the polymerization procedure at high temperature. Such species are reported to significantly contribute to the stability of the triazine structure [31].

To examine the electronic properties of the samples, EPR tests were used for characterization. As shown in Figure 2f, a significant Lorentz curve is observed in the samples with a g-factor value of 2.005, typically correlated with the existence of N defects within the carbon nitride structure. The EPR intensity of the Pt-PTI₃ sample exhibits a noticeable decrease in comparison to that observed for the Pt-PCN, suggesting a lower density of unpaired electrons. This result implies fewer structural defects and a more complete π -conjugated system, which facilitates the isolation and migration of the electric charges [39]. Furthermore, the thermal stability of the sample in an oxygen environment was examined using TGA, and the findings are illustrated in Figure S8. The decomposition of PCN and PTI samples occurred at 600 °C, indicating good thermal stability, which ensures their stability during PTC reaction.

2.2. Photoelectrochemical Properties of Catalysts

As depicted in Figure 3a, the optical absorption properties of Pt-PTI_x and Pt-PCN samples were characterized by UV-vis diffuse reflectance spectroscopy (UV-vis DRS). The absorption band edge of Pt-PTI_x appeared at approximately 400 nm, whereas that of Pt-PCN appeared at ~510 nm [31]. The increased light absorption tail of Pt-PCN in the visible spectrum could potentially be ascribed to the presence of more abundant structural defects, which further confirms the higher degree of structural polycondensation in PTI samples [52]. The bandgap energies were estimated using the Tauc method based on the plots of $(\alpha h\nu)^{1/2}$ versus $h\nu$ (Figure 3b). The bandgap values for Pt-PTI₃ and Pt-PCN were calculated to be 3.05 eV and 2.63 eV, respectively. The relatively larger bandgap of Pt-PTI₃ indicates a more ordered electronic structure with fewer defect states, which is beneficial for efficient charge transfer and contributes to the enhanced photothermal catalytic performance of the Pt-PTI₃. These results are consistent with previously reported values in the existing literature [53]. The photoelectron-hole separation ability of the catalyst was characterized by room temperature PL and TRPL. The spectrum of PL depicted in Figure 3c indicates that the fluorescence strength of Pt-PTI_x is significantly weakened compared with Pt-PCN, among which Pt-PTI₃ is the best, which indicates that the photogenerated carrier recombination of PTI is effectively suppressed. The TRPL spectrum in Figure 3d shows that the fluorescence lifetime of Pt-PTI₃ (1.80 ns) is significantly longer than that of Pt-PCN (1.44 ns), indicating a longer lifetime and more efficient separation and migration of photogenerated carriers within Pt-PTI₃. The photocurrent response and impedance spectrum can directly reveal the photoelectric response ability of the samples. The photocurrent response signal of Pt-PTI₃ in Figure 3e exhibits a significantly enhanced intensity compared to Pt-PCN, suggesting that the molten-salt-treated Pt-PTI₃ sample possesses superior conductivity. This implies efficient transmission of photogenerated carriers within the structure of Pt-PTI₃. The significantly smaller Nernst curve-ring radius of Pt-PTI₃ in Figure 3f also verifies this conclusion.

The redox capacity of a catalyst and the generation of active species are closely related to its energy-band location. To comprehend the band structure of the catalyst, the conduction band (CB) location was obtained from the Mott-Schottky curves (Figure S9). The measurements were carried out using an Ag/AgCl reference electrode, and the potentials were converted to the NHE scale according to $E_{\text{NHE}} = E_{\text{Ag/AgCl}} + 0.197 \text{ V}$. The positive

slopes of the Mott–Schottky curves indicate that both PTI_3 and PCN have typical n-type semiconductors. The test results show that the CB positions of PTI_3 and PCN are -0.67 V and -1.20 V (vs. NHE), respectively. Based on the bandgap value in Figure 3b, the PTI_3 and PCN samples exhibit valence band (VB) positions of 2.38 V and 1.43 V (vs. NHE), respectively. The results indicate that the oxidation of PTI was significantly stronger than that of PCN, thereby promoting the oxidative degradation of VOCs.

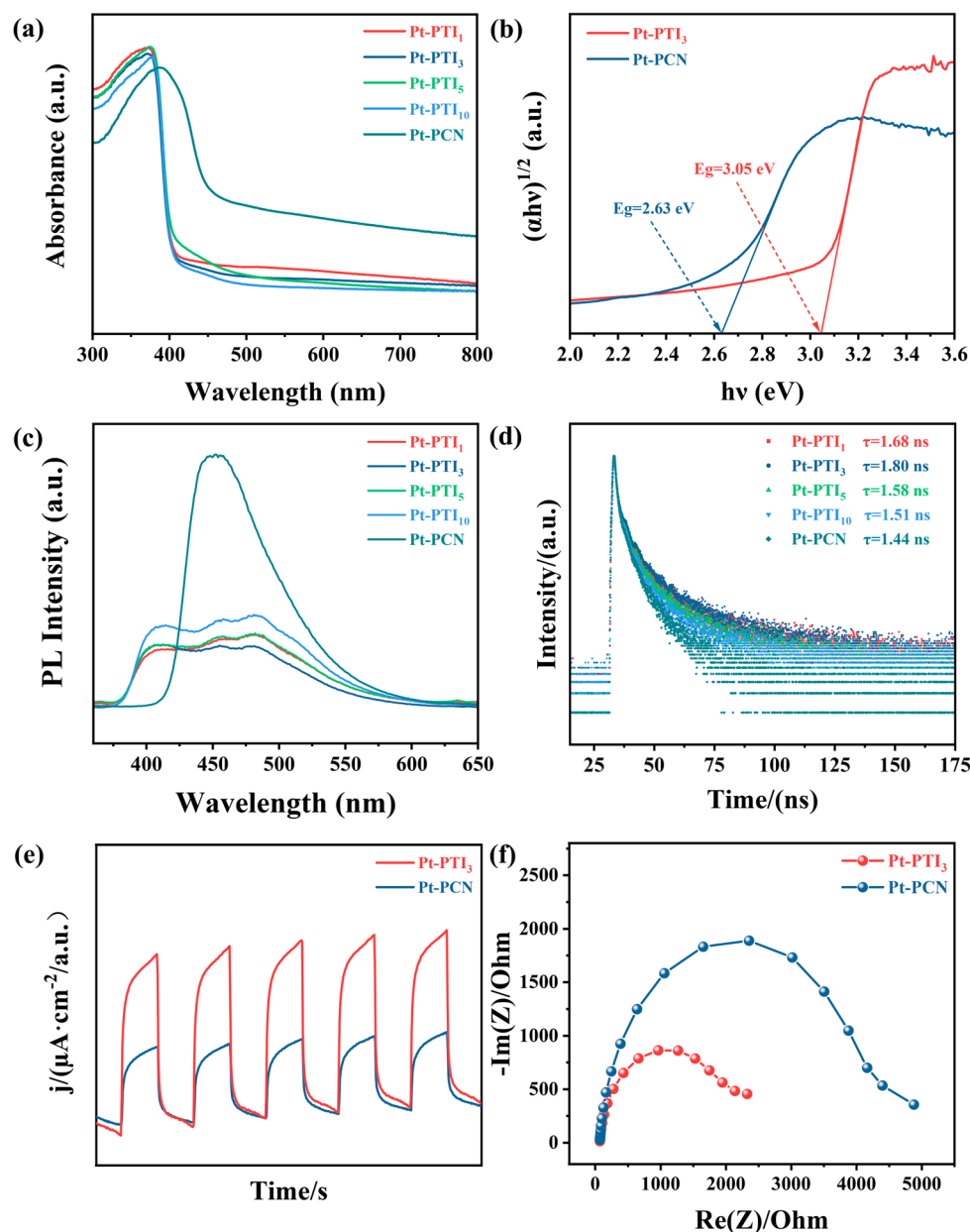


Figure 3. (a) UV–vis diffuse reflectance spectroscopy spectrum of Pt-PTI_X and Pt-PCN; (b) bandgap of Pt-PTI₃ and Pt-PCN determined by Tauc plots; (c) PL spectrum; (d) time-resolved PL spectrum of Pt-PTI_X and Pt-PCN; (e) photocurrent response; and (f) EIS Nyquist plots of Pt-PTI₃ and Pt-PCN.

During the catalytic oxidation process, the presence of electrons (e^-) and photogenerated holes (h^+) with oxidizing and reducing capabilities facilitates the conversion of adsorbed H_2O and O_2 on the catalyst surface into $\cdot\text{OH}$ and $\cdot\text{O}_2^-$ radicals, respectively. According to the above information, the VB position (2.38 eV) of Pt-PTI₃ exhibits a higher energy level compared to the hydroxyl group redox position ($\cdot\text{OH}/\text{OH}^-$, 1.89 V vs. NHE), while its CB position (-0.67 eV) demonstrates a lower energy level than the oxygen redox

position ($\text{O}_2/\cdot\text{O}_2^-$, -0.28 V vs. NHE). Theoretically, the PC process of Pt-PTI₃ materials has the potential to produce both of these active species, whereas $\cdot\text{OH}$ cannot be generated on PCN due to its lower VB position [54,55]. For this purpose, 5,5-Dimethyl-1-pyrroline-N-oxide (DMPO) was applied as the trapping agent in EPR techniques to confirm the generation of $\cdot\text{O}_2^-$ and $\cdot\text{OH}$ species from Pt-PTI₃ and Pt-PCN samples. According to Figure 4a,b, no corresponding EPR signals were detected from either of the samples under conditions of darkness. After 10 min of illumination, the Pt-PTI₃ catalyst exhibited the characteristic EPR signals corresponding to DMPO- $\cdot\text{O}_2^-$ and DMPO- $\cdot\text{OH}$, while only the DMPO- $\cdot\text{O}_2^-$ signal was observed in Pt-PCN, and its intensity was significantly weaker than that of Pt-PTI₃, which is consistent with the band structure analysis. This may be due to the more efficient photogenic charge transfer capability of PTI samples [56].

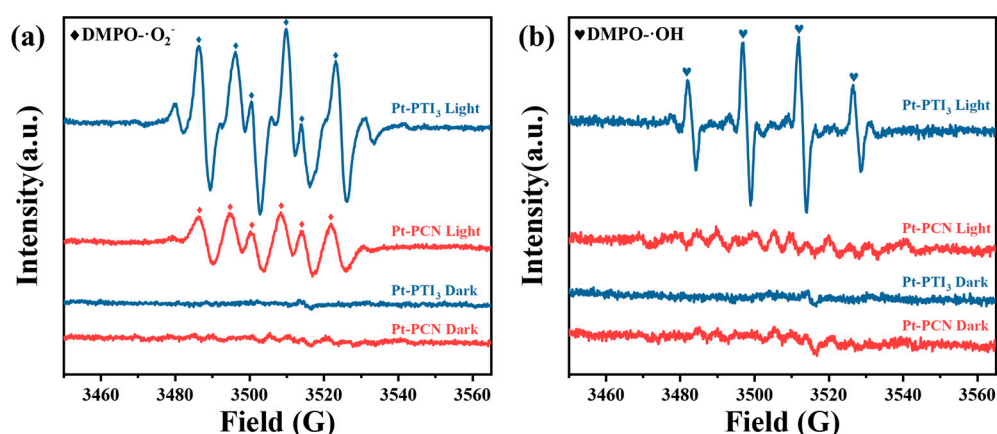


Figure 4. (a) DMPO- $\cdot\text{OH}$ and (b) DMPO- $\cdot\text{O}_2^-$ EPR spectrum of Pt-PTI₃ and Pt-PCN in the dark or under illumination.

2.3. Photothermal Catalytic Performance on Toluene Oxidation

The PTC performance was assessed in a sequential flow system with toluene (250 ppm). The effect of temperature on the PTC efficiency of the catalyst in illumination conditions is illustrated in Figure 5a. Apparently, the PTC performance of all Pt-PTI_x samples surpasses that of Pt-PCN, which can realize the oxidative decomposition of toluene under milder conditions. Among them, Pt-PTI₃ exhibits the highest level of activity, enabling complete toluene degradation at a temperature of 143 °C, while Pt-PCN requires 178 °C under the same conditions. It can be noticed that at 120, 130 and 140 °C, the conversion rate of Pt-PTI₃ reached 35.3%, 79.9% and 96.6%, respectively, while at the same temperature, that of Pt-PCN only increased 5.8%, 13.4% and 25.6%, with 5.09, 4.96 and 2.77 times, respectively. To assess the synergistic effect of PTC, we conducted additional investigations on the conversion rate and mineralization rate of the Pt-PTI₃ sample under both photothermal and thermal conditions. The rates of conversion and mineralization were found to be higher under photothermal conditions compared to thermal-only conditions at the same reaction temperature in Figure 5b,c, in which the fully converted temperature was at 143 °C in the photothermal conditions and 162 °C for the thermal-only condition. The degree of mineralization of toluene degraded by Pt-PTI₃ is remarkable, reaching 87.5% at 150 °C based on the production of CO₂ during the reaction, indicating a high degree of oxidation of toluene. Clearly, the photothermal effect of light makes the catalysts behave better in the PTC reaction [8].

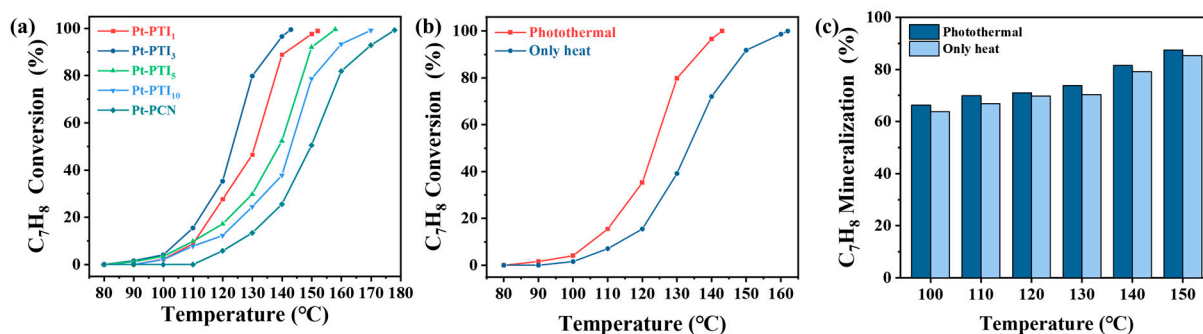


Figure 5. (a) Photothermal catalytic efficiency on toluene conversion at different temperatures of Pt-PTI_x and Pt-PCN; (b) photothermal catalytic/thermocatalytic efficiency on toluene conversion; and (c) photothermal catalytic/thermocatalytic efficiency on toluene mineralization of Pt-PTI₃.

In addition, the stability of Pt-PTI₃ in continuous PTC degradation of toluene, as well as cycling tests was also investigated. As shown in Figure 6a, Pt-PTI₃ can achieve almost complete degradation of toluene within 1 h in the continuous 24 h degradation test, and no deactivation of the catalyst occurs during the 24 h test. The conversion rate is still maintained at 98–100%, and the mineralization rate is stable between 79 and 88 cyclic test shown in Figure 6b indicates that Pt-PTI₃ exhibits no significant deactivation after five cycles. As depicted in Figure S10, the XRD and FT-IR spectra of the samples before and after the cyclic test showed comparable patterns, which proved that the crystal structure and chemical composition of Pt-PTI₃ did not change, and the structural and morphological stability before and after the reaction was excellent. In practical industrial applications, the concentration of VOCs is not limited to a fixed concentration or type. Therefore, we also investigated the degradation performance of Pt-PTI₃ samples against a series of gradient concentrations (250, 500, 750, and 1000 ppm) of toluene (Figure S11). The results showed that Pt-PTI₃ demonstrates excellent PTC performance over a wide range of toluene concentrations. According to Figure S12, Pt-PTI₃ material also has excellent PTC degradation performance for other aromatic VOCs (such as benzene).

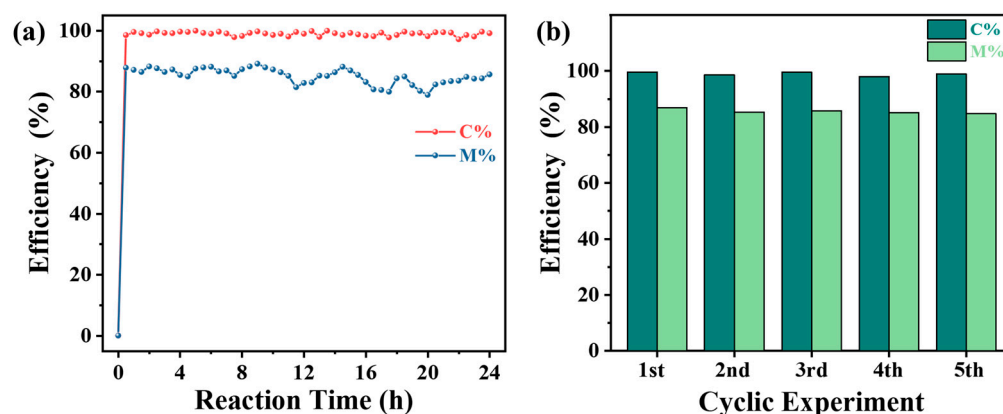


Figure 6. Photothermal catalytic performance on toluene conversion and mineralization of Pt-PTI₃: (a) long-time (24 h) tests; (b) five-cycle tests. Here, C represents the toluene conversion, and M represents the mineralization rate calculated based on the CO₂ production during the reaction.

2.4. Photothermal Reaction Mechanism and Synergistic Effect

Combined with the findings of the above characterization and analysis, we suggest a possible PTC reaction mechanism. VOCs are oxidized by redox reactions with holes (h⁺) and electrons (e[−]) generated by the photoexcitation process, and can also react with the ·OH and ·O₂[−] free radicals [57]. The ·OH and ·O₂[−] generated by the PC reaction promote effective activation and deep oxidation of toluene. The sustained supply of energy for

the TC process is supported by the light-induced photothermal effect and the addition of thermal energy, which promotes the effective migration and separation of photogenerated charges. Specifically, upon exposure to light and heat, photogenerated electrons (e^-) originating from semiconductor carriers are rapidly transferred to Pt nanoparticles, which act as electron sinks and promote charge separation. According to the XPS analysis, Pt mainly exists in the metallic state (Pt^0), which is beneficial for electron transfer and oxygen activation on the catalyst surface. The adsorbed O_2 on the Pt surface captures these electrons and produces $\cdot O_2^-$ radicals. With the increase in temperature, $\cdot O_2^-$ further dissociates into $[O_2^{2-}]_{ads}$ and $[O^-]_{ads}$ [57–59]. Subsequently, they engage in the oxidation of toluene. Therefore, the elevation of temperature within the catalytic system progressively enhances the consumption of photoinduced charges, thus improving the efficiency of separation and utilization of the photogenerated charges. At the same time, the oxidizing h^+ generated by photoexcitation reacts with the adsorbed H_2O to form $\cdot OH$ radical. These active species, together with h^+ , contribute to the activation and degradation of toluene molecules into CO_2 and H_2O .

3. Materials and Methods

3.1. Materials

Melamine ($C_3H_6N_6$, $\geq 99.0\%$), lithium chloride ($LiCl$, $\geq 95.0\%$), potassium chloride (KCl , $\geq 99.8\%$), and chloroplatinic acid, hexahydrate ($H_2PtCl_6 \cdot 6H_2O$) were obtained from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, and used in their original state without the need for additional purification.

3.2. Catalysts Synthesis

3.2.1. Synthesis of PCN

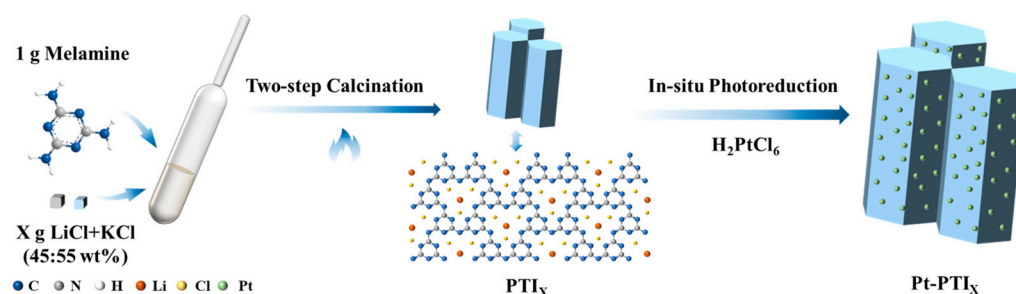
The PCN material is synthesized through a direct thermal polymerization process. The melamine (1.0 g) was placed in an alumina crucible with a lid and heated to $550\text{ }^\circ\text{C}$ in an air muffle furnace at a heating rate of $5\text{ }^\circ\text{C}/\text{min}$ for 4 h. After natural cooling, PCN powder was obtained by grinding.

3.2.2. Synthesis of PTI and Pt-PTI

As shown in Scheme 1, PTI material was synthesized using the two-step calcination method. In total, 1.0 g of melamine and X g ($X = 1, 3, 5, 10$) of a mixture of lithium chloride and potassium chloride (45:55 wt%) were thoroughly ground and filled into ampoules and calcined in a Muffle furnace at $400\text{ }^\circ\text{C}$ for 6 h. After natural cooling to room temperature, the ampoule bottle was placed in a vacuum and sealed and then calcined in a Muffle furnace at $550\text{ }^\circ\text{C}$ for 24 h. After cooling, the sample was carefully removed from the ampoule and ultrasonic dispersion with deionized water was added and washed several times to remove the residual salt in the sample. Finally, the sample was dried in a vacuum oven at $60\text{ }^\circ\text{C}$ for 12 h. The sample obtained was named PTI_X (where X is the mass of the binary salt mixture used).

Pt nanoparticles were loaded via the in situ photoreduction method. In total, 100 mg of the PTI_X sample was ultrasonically dispersed in 100 mL of deionized water, and a certain amount of H_2PtCl_6 solution (based on the content of Pt) was added to the reactor. Under the condition of continuous agitation, the gas in the reactor was removed by repeated vacuuming, and then the sample was illuminated by a 300 W xenon lamp for 1 h. At the end of the reaction, the sample was taken out and pumped, filtered, washed and dried, and the obtained sample was named Pt- PTI_X . The preparation method of the Pt-PCN sample is consistent with the above steps.

The sample was subjected to ICP-OES analysis to determine the precise quantity of Pt present. Approximately 10 mg of the catalyst was digested in 1 mL of concentrated H_2SO_4 at $150\text{ }^\circ\text{C}$, followed by tenfold dilution with deionized water and filtration through a $0.45\text{ }\mu\text{m}$ membrane prior to analysis. The corresponding data can be found in Table S1.



Scheme 1. Synthesis method of Pt-PTI_x catalysts.

3.3. Catalysts Characterization

A range of characterization techniques can be employed to assess the physical and chemical properties of catalysts. For detailed instrument models and parameters, please refer to the Supporting Information.

3.4. Catalytic Performance Evaluation

The catalytic efficiency of the samples was tested in a continuous flow PTC experimental apparatus, as shown in Figure S1, and specific details regarding the experimental conditions and methods of evaluation can be found in the accompanying Supporting Information.

4. Conclusions

In summary, the synthesis of PTI materials with enhanced crystallinity was successfully achieved through a distinctive molten salt method, employing varying ratios. The optimal ratio of the precursor to molten salt was 1:3; using the appropriate amount of molten salt can lead to the development of PTI materials with relatively smaller particle size, better dispersion, and a larger specific surface area. In addition, low-content (0.31 wt%) Pt nanoparticles were loaded using in situ photoreduction technology. The material exhibited excellent performance during the PTC oxidation of toluene at a mild temperature of $143\text{ }^\circ\text{C}$, achieving nearly 100% conversion of toluene at $143\text{ }^\circ\text{C}$, which is $35\text{ }^\circ\text{C}$ lower than that required for traditional PCN. Furthermore, the optimal mineralization rate of toluene by Pt-PTI reached 87%. According to the characterization analysis, the improved activity of the PTI material was due to the presence of more active sites, fewer structural defects, better charge transfer ability and stronger redox ability. During open-loop degradation of toluene e^- , h^+ and reactive oxygen species ($\cdot\text{OH}$ and $\cdot\text{O}_2^-$) produced during photocatalysis become involved, forming synergistic effects with the thermal catalytic reaction. This work deeply analyzes the structures and properties of the PCN catalyst, reveals the key factors of the PTC reaction, and opens up new paths for utilizing polymeric semiconductors in energy conversion and environmental governance.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/catal16040295/s1>. Figure S1: Schematic diagram of catalysts performance evaluation. Figure S2: SEM image of Pt-PCN. Figure S3: (a,b) TEM image of Pt-PCN (c) HAADF-STEM image and EDX elemental mapping of Pt-PCN sample. Figure S4: SEM image of (a) Pt-PTI₁ (b) Pt-PTI₃ (c) Pt-PTI₅ (d) Pt-PTI₁₀. Figure S5: (a) N_2 adsorption–desorption isotherms of Pt-PTI_x and Pt-PCN; (b) Pore size distribution diagram of Pt-PTI_x and Pt-PCN. Figure S6: Solid-state ^{13}C NMR spectrum of PTI₃. Figure S7: (a) Raman spectra of Pt-PTI_x and Pt-PCN; XPS spectra

of (b) Li 1s and (c) Cl 2p of the Pt-PTI₃ sample. Figure S8: TGA spectra of Pt-PTI₃ and Pt-PCN. Figure S9: (a) Mott–Schottky curve of the PTI₃ sample measured using an Ag/AgCl reference electrode; (b) Mott–Schottky curve of the PCN sample. The calculated conduction band (CB) positions are -0.67 V and -1.20 V (vs. NHE), respectively. Figure S10: (a) XRD patterns of Pt-PTI₃ before and after reaction; (b) FTIR spectra of Pt-PTI₃ before and after reaction. Figure S11: Photothermal catalytic performance of Pt-PTI₃ for toluene at different concentrations. Figure S12: Photothermal catalytic performance on benzene conversion at different temperatures (250 ppm benzene, 300 mW/cm² Xe lamp, WHSV = 12,000 mL/(g·h)) of Pt-PTI_x and Pt-PCN. Table S1: Chemical compositions of the samples obtained from ICP; Table S2: Specific surface area, pore volume and pore size of Pt-PTI_x and Pt-PCN.

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