Research Article

Removal of Formaldehyde Using Highly Active Pt/TiO$_2$ Catalysts without Irradiation

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Formaldehyde (HCHO) is one of the major indoor air pollutants. TiO$_2$ supported Pt catalysts were prepared by sol-gel method and used to eliminate HCHO at room temperature without irradiation. The reduced Pt/TiO$_2$ catalyst (denoted as Pt/TiO$_2$-H$_2$) showed much higher activity than that calcined in air (denoted as Pt/TiO$_2$-air). More than 96% of the conversion of HCHO was obtained over 0.5 wt% Pt/TiO$_2$-H$_2$, on which highly dispersed metallic Pt nanoparticles with very small size (~2 nm) were identified. Metallic Pt rather than cationic Pt nanoparticles provide the active sites for HCHO oxidation. Negatively charged metallic Pt nanoparticles facilitate the transfer of charge and oxygen species and the activation of oxygen.

1. Introduction

Gaseous formaldehyde (HCHO) may cause adverse effects on both air quality and human health [1–3]. Great efforts have been made to eliminate indoor HCHO to satisfy the stringent environmental regulations and improve air quality. Physical adsorption with porous materials or the combined physical adsorption and chemical reaction by impregnating the chemical reagents (such as potassium permanganate and organic amine) on the adsorption materials was found to be effective for eliminating HCHO emission but only for a short period before saturation. The overall efficiency of these adsorbent materials is not so promising due to their limited removal capacities [4, 5]. Catalytic oxidation is recognized as the most promising HCHO removal technology [6–8]. Nevertheless, HCHO oxidation can only occur under irradiation (UV or visible light) or at high temperature over most of the reported catalysts [9–11]. In these cases, an extra irradiation or heating apparatus is needed, causing higher operating cost and more severe reaction conditions, which is not suitable for the control of indoor air pollution [10]. For air cleaning, low energy demand and low concentration of HCHO strongly require a catalyst to exhibit high activity for complete oxidation of

HCHO. HCHO purification at room temperature is highly desirable due to its environment-friendly reaction conditions and energy saving consideration [6, 7, 12]. Moreover, indoor air is often enriched with water vapor, which frequently leads to severe catalytic deactivation through the strong adsorption of water on the active sites, especially at low temperatures [10]. The development of effective catalysts for complete oxidation of low-concentration HCHO at ambient temperature is a challenging subject for scientists [4, 7].

Recently, several studies related to HCHO removal at low temperature have been carried out using supported noble metal (Pt, Rh, Au, and Pd) [6, 7, 13, 14] and nonnoble metal catalysts [15, 16]. Among them, Pt/TiO$_2$ is the most active [6, 14]. Peng and Wang [5] found that HCHO conversion for 1 wt% Pt/TiO$_2$ is only 14.3% at 20°C and it increased to 97% at 120°C. However, the oxidation state of active component may be changed due to HCHO reduction at high temperature, which may cause confusion to mechanistic study although supported Pt catalysts have been proved to be effective for HCHO oxidation at low temperature, even at room temperature in some cases. However, high loading of Pt is generally needed for effective oxidation of HCHO, which greatly limits its widespread application due to the expensive cost [10]. One
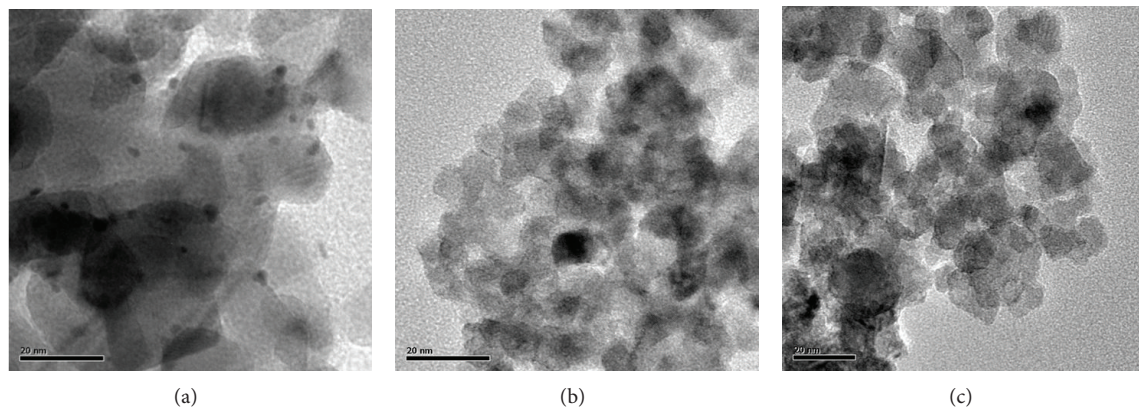
(a)   (b)   (c)

FIGURE 1: TEM micrographs for (a) Pt/TiO_2-H_2, (b) Pt/TiO_2-air, and (c) TiO_2.

alternative is to improve the catalytic activity by optimizing catalysts with lower Pt loading. In addition, several hundred ppm of HCHO concentration used for the performance test is much higher than the normal HCHO concentration in indoor environments, which generally ranges from dozens of ppb to ppm. Furthermore, the Pt catalysts were generally pretreated in air at high temperature [4, 6, 7, 14], which result in the formation of PtOx. As known, oxidation state of Pt is one of the most important factors controlling the catalytic activity [17]. The effect of metallic Pt on catalytic activity and the mechanism leading to high activity for HCHO oxidation at room temperature has not well demonstrated yet.

Herein, we present an effective catalyst with low Pt loading for the purification of indoor HCHO at room temperature without any irradiation. Efficient elimination of low-concentration HCHO was obtained over a reduced 0.5 wt% Pt/TiO_2 catalyst prepared by sol-gel method. Structural analysis of the catalysts used was performed and subsequently correlated with their catalytic performances to investigate the mechanism leading to the high activity over the reduced Pt nanoparticles.

2. Experimental Sections

2.1. Preparation and Characterization of Pt/TiO_2 Catalysts. The Pt-doped TiO_2 catalysts were prepared by the sol-gel method. A mixture solution containing 34 mL of ethanol, 1.7 mL of water, and 1.7 mL of hydrochloric acid was added dropwise under vigorous stirring to another mixture solution containing 17 mL of tetrabutyl titanate, 5.1 mL of 0.02 mol/L chloroplatinic acid ($H_2PtCl_6 \cdot 6H_2O$), and 45 mL of absolute ethanol. The resulting transparent colloidal suspension was stirred for 5 h and aged until the formation of gel. The gel was dried at 353 K for 12 h and then calcined at 673 K for 4 h in air and hydrogen flow, obtaining the oxidized and reduced Pt catalysts, respectively. They were referred to as Pt/TiO_2-air and Pt/TiO_2-H_2, respectively. The Pt loading is 0.5 wt% on both catalysts. The catalysts were crushed and then sieved through 40–60 meshes.

Transmission electron microscopy (TEM) images were recorded by a Tecnai G2 20 microscope operated at 200 kV.

BET surface areas of the samples were measured by N_2 adsorption-desorption isotherms at 77 K using a Micromeritics ASAP 2020 instrument. Prior to the measurement, the samples were degassed at 573 K for 2 h. X-ray powder diffraction (XRD) patterns were collected with a Bruker D8 Advance X-ray powder diffractometer, using Cu Kα (λ = 1.5418 Å) radiation. The working voltage of the instrument was 40 kV, and the current was 40 mA. The intensity data were collected in a 2θ range from 20° to 90°. The average crystal size was determined from the anatase peak broadening (101) crystal plane for anatase with Scherrer's formula. X-ray photoelectron spectroscopy (XPS) measurements of the catalysts were performed with a PHI 5000 VersaProbe system using a monochromatic Al Kα source. The binding energies (BEs) were determined by utilizing C1s line as a reference with energy of 284.8 eV.

2.2. Measurement of Catalytic Activity. The oxidation of HCHO was performed in a quartz tubular (i.d. = 6 mm) fixed-bed reactor in a dark environment at ambient temperature (25 ± 1°C). Also, 0.5 g of the catalyst in 40–60 mesh was loaded in the reactor. Gaseous HCHO was generated by zero air flowing through an HCHO solution in an incubator. An air mixture containing 10 ppm HCHO and water vapor (50% relative humidity) was introduced as the reactants. The total flow rate was 1 L/min, corresponding to a gas hourly space velocity (GHSV) of 120,000 mL/g_{cat}·h. The HCHO and CO_2 in the air stream were analyzed by an HCHO monitor (Formaldemeter 400, PPM Technology) and a CO_2 monitor (HAL-HCO$_2$01, Chinaway), respectively.

3. Results and Discussion

3.1. Catalyst Characterization

3.1.1. TEM. Representative TEM micrographs of the TiO_2 and Pt/TiO_2 are presented in Figure 1. The TEM micrograph for the Pt/TiO_2-air catalyst was similar to that of TiO_2. No distinguishable Pt particles are observed over the Pt/TiO_2-air catalyst, indicating that their sizes were probably very small and could not be detected by TEM. Pt nanoparticles prepared

TABLE 1: BET surface area of Pt/TiO_2 and TiO_2.

Sample	Pt/TiO_2-H_2	Pt/TiO_2-air	TiO_2	P-25
BET surface area (m^2/g)	74.1	84.5	74.9	50 [18]
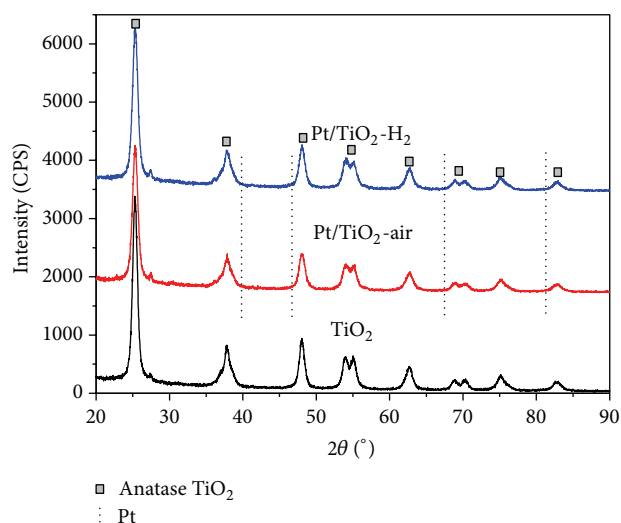
FIGURE 2: XRD patterns of the Pt/TiO_2 and TiO_2 catalysts.

by different methods existed with different crystal structure. Pt nanoparticles with very small and homogeneous size (~2 nm) present uniformly on the surface of the Pt/TiO_2-H_2 catalyst. Hence, the H_2 reduction promoted the formation of Pt clusters.

3.1.2. BET Surface Areas. Table 1 shows the BET surface area of the tested catalysts. TiO_2 prepared by sol-gel method obtained a BET surface area of 74.9 m^2/g, which is much larger than that of commercially available P-25 TiO_2 (Degussa, Germany) with a BET surface area of 50 m^2/g [18]. It is worth noting that the BET surface area of Pt/TiO_2-air is increased to 84.5 m^2/g, which is larger than that of Pt/TiO_2-H_2 (74.1 m^2/g). This is probably because Pt nanoparticles on Pt/TiO_2-H_2 cover the surface of TiO_2 and block its pores. As observed in TEM, small Pt nanoparticles were highly dispersed on the support, which may cause the loss of surface area. Pt/TiO_2 prepared by sol-gel method possesses much higher surface area, which can provide more absorption sites for HCHO.

3.1.3. XRD. As shown in Figure 2, XRD patterns for pure TiO_2 exhibited strong diffraction peaks at 25.2° and 48.1° indicating TiO_2 in the anatase phase. The peaks of the Pt/TiO_2-H_2 and Pt/TiO_2-air catalyst are essentially the same as pure TiO_2, and no significant Pt peaks are observed, suggesting that Pt particles are very small, as observed by the TEM (Figure 1). The average particle sizes as calculated with the Scherrer formula were 14.1, 12.8, and 12.9 nm for the samples of TiO_2, Pt/TiO_2-H_2, and Pt/TiO_2-air, respectively. They are much smaller than P-25 with an average crystal sizes of about 30 nm [18]. The particle sizes for the Pt/TiO_2-H_2 and
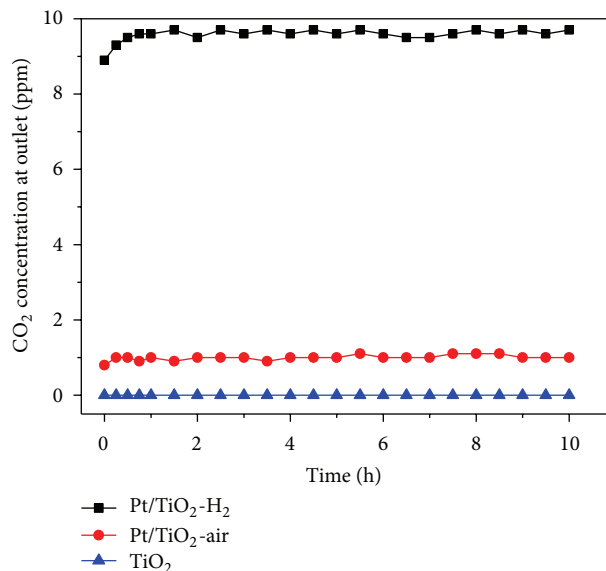
FIGURE 3: CO_2 concentration at the outlet of reactor.
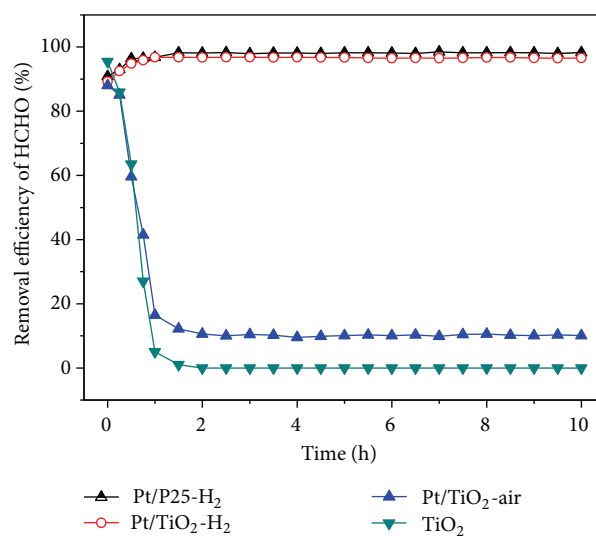
FIGURE 4: HCHO removal efficiency over the Pt/TiO_2 and TiO_2 catalysts.

Pt/TiO_2-air are smaller than TiO_2, indicating that the deposition of Pt can inhibit the growth of TiO_2 crystal. The doping of Pt probably changed the surface character of TiO_2 and avoided the sintering and conglobation during thermal treatment hence inhibiting its growth. It can be found that the average particle size of the Pt/TiO_2-H_2 catalyst is almost the same as that of the Pt/TiO_2-air catalyst. The small TiO_2 crystal accounts for the high BET surface area of the Pt/TiO_2, as shown in Table 1.

3.2. Catalytic Activity Test. HCHO oxidation by pure TiO_2 can be excluded from the discussion since no CO_2 was identified during the reaction, as shown in Figure 3. However, CO_2 concentration was increased to about 1 ppm and 9.6 ppm over Pt/TiO_2-air and Pt/TiO_2-H_2, respectively. Figure 4 shows
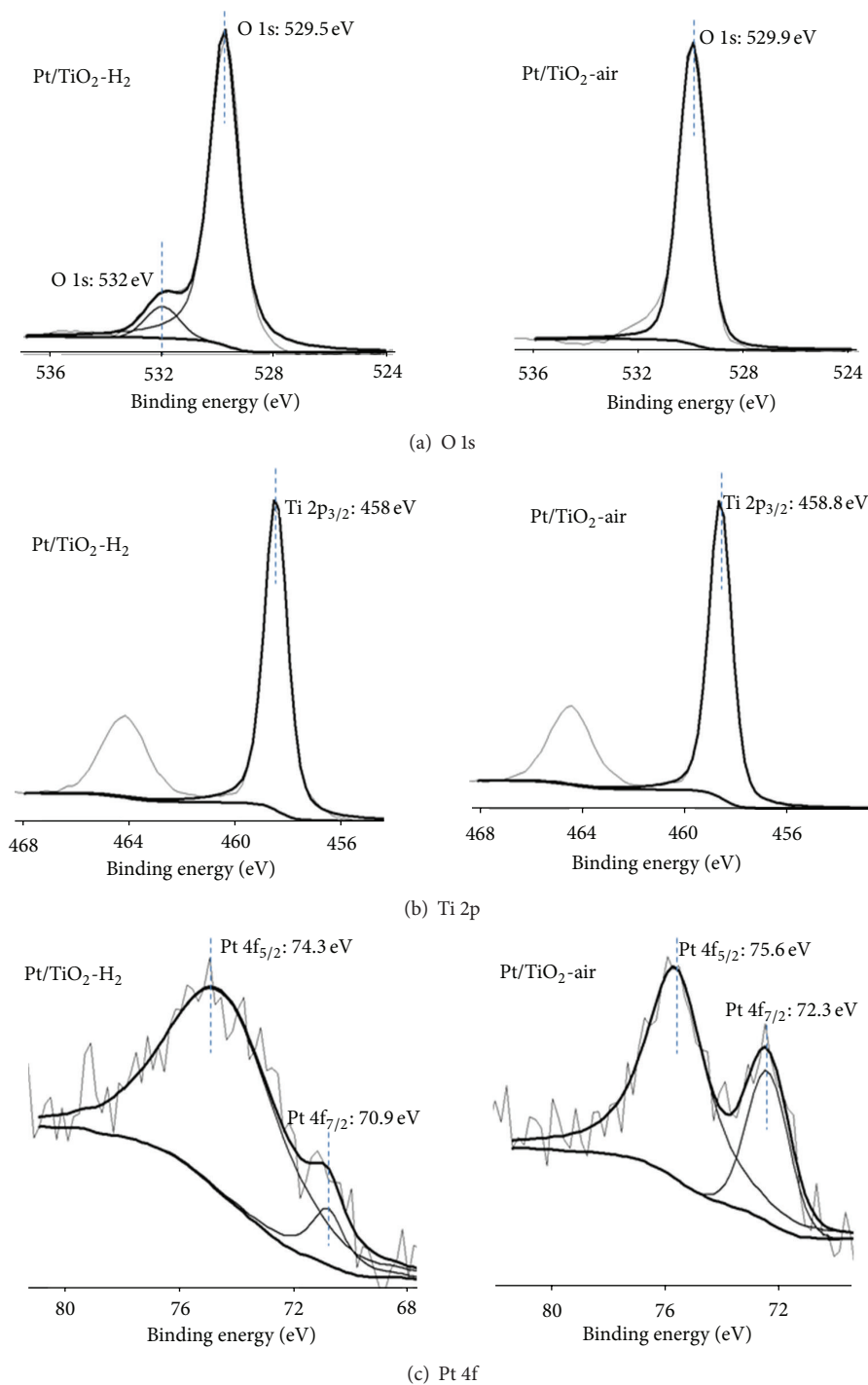
(a) O 1s

(b) Ti 2p

(c) Pt 4f

FIGURE 5: XPS spectra of the Pt/TiO$_2$-H$_2$ and Pt/TiO$_2$-air catalyst.

the time dependence of HCHO removal efficiency for Pt/TiO$_2$ together with TiO$_2$ at ambient temperature. The removal of HCHO at initial stage over pure TiO$_2$ and Pt/TiO$_2$-air was mainly ascribed to the adsorption by TiO$_2$. Only about 10% HCHO conversion was obtained over the Pt/TiO$_2$-air catalyst at the stable stage, whereas it was notably increased to 96.6% over the Pt/TiO$_2$-H$_2$ catalyst. HCHO removal efficiency was significantly improved over the Pt/TiO$_2$-H$_2$ catalyst.

The results showed that the catalytic activities were greatly influenced by the catalyst's preparation methods.

The most important factors controlling the catalytic activity of nanoscale noble metal catalysts appear to be the particle size, nature of support, and noble metal oxidation state [19]. The smaller the size of nanoparticles is, the more active centers are formed, leading to better catalytic activity [20]. As described previously, the unreduced catalyst possesses higher

surface area and smaller Pt particles than the reduced one, which can provide more active sites for the adsorption and oxidation of HCHO. Therefore, the Pt/TiO$_2$-air catalyst is expected to be more active for HCHO oxidation than the Pt/TiO$_2$-H$_2$ catalyst. On the contrary, the former showed much poorer activity for HCHO oxidation than the latter, as shown in Figure 4. Therefore, Pt oxidation state should be the most important factors controlling the catalytic activity in this reaction. Reduced Pt supported on commercial TiO$_2$ (P25, Degussa) (Pt/P25-H$_2$) also confirmed this. HCHO removal efficiency reached 98% over 0.5% Pt/P25-H$_2$. Pt oxidation state was analyzed by XPS, as shown in Figure 5(a). The binding energy of Pt 4f$_{7/2}$ is 72.3 and 70.9 eV on the Pt/TiO$_2$-air and Pt/TiO$_2$-H$_2$ catalyst, corresponding to Pt^{2+} and Pt^0 [21], respectively. Pt was reduced into metallic state on the Pt/TiO$_2$-H$_2$ catalyst. Figure 4 clearly showed that the reduced Pt/TiO$_2$ with metallic Pt achieved much higher HCHO removal efficiency than the oxidized one with Pt^{2+}, suggesting that metallic Pt is the active site for HCHO oxidation. Similar conclusions were made for active centers of Pd [22–25] and Au [26–28] in oxidation reaction. It is worth noting that Pt 4f$_{7/2}$ of Pt/TiO$_2$-H$_2$ was shifted to lower BE by approximately 0.3 eV compared with bulk metallic Pt with typical Pt 4f$_{7/2}$ BE around 71.2 eV, indicating that nanoparticles are negatively charged on the Pt/TiO$_2$-H$_2$ catalyst. The electron transfer was proposed to be responsible for this negative shift [21]. It can be observed from Figure 5(b) that Ti 2p$_{3/2}$ on the Pt/TiO$_2$-air catalyst is located at 458.8 eV, corresponding to Ti^{4+} of TiO$_2$. However, it was slightly shifted to 458.4 eV on the Pt/TiO$_2$-H$_2$ catalyst, suggesting that Ti^{4+} is partially reduced. The electron transfer can be expected from the reduced TiO$_2$ to Pt nanoparticles [29]. Oxygen vacancies on the reduced TiO$_2$ and the negative charged Pt showed enhanced capacity of O$_2$ adsorption. The charge was further transferred from negative charged Pt to the adsorbed O$_2$, leading to negative charge of adsorbed O$_2$. This was confirmed by the change of O 1s BE after reduction, as shown in Figure 5(c). The O 1s core level peak on the Pt/TiO$_2$-air catalyst appears at 529.9 eV, which is ascribed to the lattice oxygen in TiO$_2$ [30]. However, it was negatively shifted to 529.5 eV on the Pt/TiO$_2$-H$_2$ catalyst. Charge transfer from Pt particles to oxygen in the meanwhile activates the oxygen. Activated oxygen may exist in the form of Pt-O complex. It was confirmed by a significant shoulder peak of O 1s at 532.0 eV on the Pt/TiO$_2$-H$_2$ catalyst, as shown in Figure 5(c). This shoulder peak of O 1s should belong to O of OH groups on the surface of the catalyst [31]. It is probably highly active and responsible for the HCHO oxidation. Thus, the transfer of charge and oxygen species and the activation of oxygen can efficiently occur at room temperature, which may account for the high activity of the Pt/TiO$_2$-H$_2$ catalyst for HCHO oxidation.

HCHO in indoor air is often enriched with water vapor, which frequently leads to severe catalytic deactivation through the strong adsorption on the active sites, especially at low temperatures [10]. The Pt/TiO$_2$-H$_2$ catalyst can keep active in moist air (50% relative humidity) at room temperature, and no deactivation was observed after reaction for 10 h. Thus, the activity of Pt/TiO$_2$ is stable and resistant to water.

4. Conclusions

Purification of indoor HCHO at room temperature was investigated using TiO$_2$ supported Pt catalysts without irradiation. The H$_2$ reduced Pt/TiO$_2$ catalysts showed much higher removal efficiency than the unreduced one. More than 96% HCHO conversion was obtained over the reduced 0.5 wt% Pt/TiO$_2$ catalyst, on which highly dispersed metallic Pt nanoparticles with very small size (~2 nm) were identified. Oxidation state of Pt is the most important factor controlling the catalytic activity. Metallic Pt rather than cationic Pt nanoparticles provide the active sites for HCHO oxidation. Efficient purification of indoor HCHO was successfully achieved over a low Pt-loading catalyst at room temperature without any heating or illumination. It is a very efficient and promising technology for the removal of indoor HCHO.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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