

# Effect of Preparation Methods on the Performance of Pt/CeO<sub>2</sub> Catalysts for the Catalytic Oxidation of Carbon Monoxide

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**Abstract** A series of  $Pt/CeO_2$  catalysts were prepared by impregnation (IM), deposition-precipitation (DP) and impregnation-reduction (IR) methods, and their catalytic activities for the oxidation of CO were evaluated at room temperature.  $Pt/CeO_2$  (IR) catalyst presented the best catalytic activity, over which CO could be completely oxidized at room temperature. Based on the characterization, it was found that on  $Pt/CeO_2$  (IR) catalyst more negatively charged metallic Pt species existed, which could efficiently absorb atmospheric oxygen and thus enhance the CO oxidation.

#### **Graphical Abstract**



**Keywords**  $CO \cdot Pt/CeO_2 \cdot Preparation method$ 

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# **1** Introduction

Carbon monoxide (CO), as a typical inflammable and explosive gaseous contaminant, seriously threatens the safety of human. Moreover, long-term exposure to CO leads to harmful effects on human health [1]. Therefore, elimination of CO has attracted more and more attention in many aspects, such as  $CO_2$  lasers, fuel cells, indoor air cleaning, automotive exhaust treatment and so on [2]. Different methods have been put into the removal of CO, for example, the installation of ventilation equipment and adsorbents with large pores. Among them the catalytic oxidation has been regarded as a potential and green technology for the removal of CO [3].

Recently, great progress has been made in the catalytic oxidation of CO and two types of catalyst, including metal oxide and noble metal catalyst, have been applied for the oxidation of CO. Previous studies have demonstrated that Cu<sub>x</sub>O/CeO<sub>2</sub> catalyst is a potential candidate for the elimination of CO. 100% CO conversion can be achieved in the temperature range of 100–140 °C [4–9]. Over Ag/CeO<sub>2</sub> [10] and CuO/Ti<sub>x</sub>Zr<sub>1-x</sub>O<sub>2</sub> [11] catalysts, CO can be completely removed at 200 and 150 °C, respectively. These facts indicate that high temperature is needed for the complete conversion of CO on metal oxide catalysts, which limited the application of this type of catalyst [1]. In contrast, noble catalysts could effectively eliminate CO at low temperature [12–18] and Au catalyst has been proved to be the most active one. CO could be completely removed at room temperature over Au/γ-Al<sub>2</sub>O<sub>3</sub> [12], Au/TiO<sub>2</sub> [14], Au/Fe<sub>2</sub>O<sub>3</sub> [15] and Au/CeO<sub>2</sub>-TiO<sub>2</sub> [18], respectively.

Different from Au, Pt catalyst was not active for the CO oxidation due to the CO self-poisoning effect. Pt active sites are occupied by the presence of CO due to its high sticking coefficient [19, 20]. In that case,  $O_2$  could not be adsorbed on Pt active sites according to the Langmuir–Hinshelwood

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mechanism [21, 22], leading to the deactivation of Pt catalyst for the CO oxidation. Bera et al. have reported that CO could be removed over 2% Pt/CeO2 at 150°C with a GHSV of  $30,000 \text{ h}^{-1}$  [23]. Liu et al. have found that Pt group metal catalysts operated well in a wide temperature window (80-180 °C) for the preferential oxidation of CO [24]. Pt<sub>30</sub>Ni<sub>22</sub>Co<sub>30</sub> nanoalloy supported on three different supports have been investigated and CO could be completely removed at 55 °C on Pt<sub>39</sub>Ni<sub>22</sub>Co<sub>39</sub>/TiO<sub>2</sub> catalyst [25]. This research showed that the activity is dependent on the type of the support. CeO<sub>2</sub> has been recognized as an attractive support due to its unique redox properties. Ce3+ ions could facilitate the adsorption and activation of O<sub>2</sub> [26], and also enhance oxygen transfer to active species [27]. Chen et al. reported that Au/CeO<sub>2</sub> catalyst with more Ce<sup>3+</sup> ions presented higher activity for the oxidation of formaldehyde [28].

Preparation method could significantly influence on the catalytic properties of catalysts. It has been found that precipitant affected the size and chemical state of Au species [28]. Na et al. [29] have found that compared with impregnation method and deposition-precipitation method, impregnation deposition-precipitation method could afford separated Pt active sites and Au active sites, thus promoting the activity of the catalyst. Huang et al. found that the Pt catalyst prepared by reduction method showed excellent catalytic activity for the oxidation of formaldehyde [30]. Herein, Pt/CeO<sub>2</sub> catalysts were prepared by impregnation (IM), deposition-precipitation (DP) and impregnation-reduction (IR) methods, and their catalytic activities for the oxidation of CO were evaluated. Physicochemical properties of the catalysts have been characterized and subsequently correlated with their catalytic performance to elucidate the origin of the high CO oxidation activity.

## 2 Experimental

## 2.1 Catalyst Preparation

A series of Pt/CeO<sub>2</sub> catalysts were prepared by impregnation method [donated as Pt/CeO<sub>2</sub> (IM)], deposition-precipitation method [donated as Pt/CeO<sub>2</sub> (DP)] and impregnation-reduction method [donated as Pt/CeO<sub>2</sub> (IR)]. The theoretical content of Pt in all the catalysts is 1 wt%. Pt/CeO<sub>2</sub> (IM) catalyst was prepared as follows: CeO<sub>2</sub> support was added into H<sub>2</sub>PtCl<sub>6</sub> solution and the suspension was stirred for 2 h. After impregnation the suspension was dried at 60 °C by rotary evaporator under vacuum. The sample was dried at 120 °C for 12 h and then treated at 400 °C in 5 % H<sub>2</sub>/N<sub>2</sub> for 6 h to obtain Pt/CeO<sub>2</sub> (IM) catalyst.

 $Pt/CeO_2$  (DP) catalyst was prepared as follows:  $Ce(NO_3)_2$  was added to  $H_2PtCl_6$  solution to form a homogeneous system and the pH value of the system was adjusted to 10 by

NaOH solution with vigorously being stirred. The system was aged at 70 °C for 2 h and then filtered and washed with distilled water to remove chlorine anion. The residue was dried at 120 °C for 12 h and then treated at 400 °C in 5%  $H_2/N_2$  for 6 h to obtain Pt/CeO<sub>2</sub> (DP) catalyst.

Pt/CeO<sub>2</sub> (IR) catalyst was prepared as follows: Ce(NO<sub>3</sub>)<sub>2</sub> was added to H<sub>2</sub>PtCl<sub>6</sub> solution to form a suspension, then NaBH<sub>4</sub> solution was quickly added into the suspension (NaBH<sub>4</sub>/Pt=20, molar ration) with vigorously being stirred. After being stirred for 2 h, the suspension was filtered and washed with distilled water to remove chlorine anion. The residue was dried at 80 °C for 12 h under vacuum to obtain Pt/CeO<sub>2</sub> (IR) catalyst.

## 2.2 Catalyst Characterization

Surface areas of the catalysts were determined by the BET method by a Micromeritics ASAP 2000 instrument. The surface chemical states of Pt/CeO2 catalysts were investigated by XPS (PHI Quantro SXM ULVAC-PHI, Japan) using an Al Ka X-ray source (1486.7 eV) at 15 kV and 25 W with the binding energy calibrated by C1s at 284.8 eV. X-ray powder diffraction (XRD) patterns were recorded with a Shimadzu XRD-6000 diffractometer operated at 40 kV and 40 mA, using nickel-filtered Cu K $\alpha$  ( $\lambda$ =0.1542 nm) radiation. High-resolution transmission electron microscopy (HRTEM) micrographs were obtained with a Tecnai G2 20 S-TWIN microscope and operated at 200 kV. CO chemisorption measurements were measured by an Autochem II 2920 automated chemisorption analyzer. The reoxidation ability of the Pt/CeO<sub>2</sub> catalysts was investigated through an O2-reoxidation experiment. Catalyst was pretreated by Ar at 120 °C for 30 min. After the catalyst was cooled down to room temperature, 1% O<sub>2</sub>/N<sub>2</sub> was introduced continuously and the concentration of O2 was monitored by the Thermal Conductivity Detector (TCD).

In situ Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) spectra were recorded in a Nicolet 6700 FTIR spectrometer equipped with a MCT detector. Before experiments, catalysts were placed in a DRIFT cell equipped with ZnSe windows and were pretreated at 120 °C for 1 h by Ar at a flow rate of 100 mL min<sup>-1</sup> and then cooled to 25 °C. Subsequently, the reactant gas mixture (100 ppm CO and N<sub>2</sub>) was introduced into the DRIFT cell for 50 min at room temperature via separate mass flow controllers at a flow rate of 100 mL min<sup>-1</sup>. After that air was introduced into the DRIFT cell for 50 min at room temperature. All spectra were recorded by accumulating 32 scans with a resolution of 4 cm<sup>-1</sup>.

## 2.3 Evaluation of Catalytic Performance

The activity evaluation for the oxidation of CO was performed in a continuous flow fixed-bed quartz reactor (i.d. = 10 mm) by using 0.36 g catalyst at 25 °C. Before the activity evaluation, the Pt/CeO<sub>2</sub> catalysts were pretreated at 120 °C for 1 h by Ar at a flow rate of 100 mL min<sup>-1</sup>. After cooled to 25 °C, the gas was switched to the reaction gas consisted of 200 ppm CO and room air (50% relative humidity, RH) from air compressor as the balance gas. The total flow rate was fixed at 350 mL min<sup>-1</sup>, corresponding to a GHSV of 60,000 h<sup>-1</sup>. The concentrations of CO and CO<sub>2</sub> were monitored by a gas chromatograph (GC) equipped with Ni catalytic converter.

## **3** Results and Discussion

Figure 1 shows the activities of Pt/CeO<sub>2</sub> catalysts for the oxidation of CO under the different preparation methods. For Pt/CeO<sub>2</sub> (IM) catalyst the CO conversion is only 27% at 25 °C. The conversion increases with the increase of temperature, which could be due to the fact that the promoted desorption of adsorbed CO leaves more active sites available for the adsorption of O<sub>2</sub>. When the reaction temperature is at 70 °C, the CO can be completely removed. The Pt/CeO<sub>2</sub> (IR) catalyst presents higher active than Pt/CeO<sub>2</sub> (IM) catalyst. For Pt/CeO<sub>2</sub> (IM) catalyst, the CO conversion at 25 °C is 54% and then 100% conversion was obtained at 60 °C. In contrast, over Pt/CeO<sub>2</sub> (IR) catalyst 100% CO conversion can be achieved at room temperature. Therefore, the Pt/CeO<sub>2</sub> (IR) catalyst possesses the highest catalytic activity for the oxidation of CO at room temperature.

Figure 2 presents the effect of RH on the oxidation of CO over  $Pt/CeO_2$  (IR) catalyst at room temperature. CO conversion is 84% when the reaction gas is dry gas. CO can be completely removed when the RH is 10%. It indicates that



Fig. 1 The activities of  $Pt/CeO_2$  catalysts for the catalytic oxidation of CO

the little  $H_2O$  can improve the catalytic activity by generating OH species on the active sites, which can adsorb and oxidize the reactant. However, when the RH increases to 70%, the catalytic activity of Pt/CeO<sub>2</sub> (IR) catalyst is decreased. Previous studies have also demonstrated the negative effect of  $H_2O$  on the oxidation of CO. The oxidation of CO would be suppressed by  $H_2O$  if the  $H_2O$  content is over 200 ppm [31]. The inhibiting effect of  $H_2O$  is due to the competitive adsorption between  $H_2O$  and CO molecules on the surface twofold coordinated oxygen site [32]. The effect of Pt content on the oxidation of CO has been investigated and the results are shown in Fig. 3. It is evident that the activity of Pt/CeO<sub>2</sub> (IR) catalyst is significantly enhanced as the Pt



Fig. 2 Effect of RH on the activity of  $Pt/CeO_2$  (IR) catalyst for the catalytic oxidation of CO



Fig. 3 Effect of Pt content on the activity of  $Pt/CeO_2$  catalyst for catalytic oxidation of CO



**Fig. 4** XRD patterns of Pt/CeO<sub>2</sub> catalysts: *a* Pt/CeO<sub>2</sub> (IR) catalyst, *b* Pt/CeO<sub>2</sub> (IM) catalyst, *c* Pt/CeO<sub>2</sub> (DP) catalyst

content increased from 0.1 to 0.6 wt%. When the Pt content reaches 0.6 wt%, CO can be completely eliminated.

The XRD patterns Pt/CeO<sub>2</sub> catalysts are illustrated in the Fig. 4. It is obvious that all the catalysts present typical cubic CeO<sub>2</sub> diffraction peaks (JCPDS 43-1002). The diffraction peaks attributed to Pt species is absent, which indicates that Pt species are highly dispersed on the CeO<sub>2</sub> support. Moreover, the intensity of the diffraction peaks of Pt/CeO<sub>2</sub> (DP) catalyst becomes weaker, suggesting a decline in the crystallite size of CeO<sub>2</sub>. The dispersions of Pt species are 40.3, 46.7 and 45.8% on the Pt/CeO<sub>2</sub> (DP) catalyst, Pt/CeO<sub>2</sub> (IR) catalyst and Pt/CeO<sub>2</sub> (IM) catalyst, respectively. The Fig. 5 show that particle sizes of Pt species are 4-6, 2 and 2-6 nm on the Pt/CeO<sub>2</sub> (DP) catalyst, Pt/CeO2 (IR) catalyst and Pt/CeO2 (IM) catalyst, respectively. It can be included that the preparation methods influence the dispersions and particle size of Pt species in Pt/CeO<sub>2</sub> catalysts.



Fig. 5 HRTEM images of Pt/CeO<sub>2</sub> catalysts



Fig. 6 Pt 4f XPS spectra of Pt/CeO<sub>2</sub> catalysts: a Pt/CeO<sub>2</sub> (DP) catalyst, b Pt/CeO<sub>2</sub> (IM) catalyst, c Pt/CeO<sub>2</sub> (IR) catalyst

Table 1 Pt 4f XPS data analysis of Pt/CeO<sub>2</sub> catalysts

Catalyst	Species	BE of Pt $4f_{7/2}$ (eV)	Relative intensity (%)
Pt/CeO <sub>2</sub> (IM)	Pt <sup>0'</sup>	69.6	51.5
	$Pt^0$	71.1	48.5
Pt/CeO <sub>2</sub> (IR)	$Pt^{0'}$	69.6	95.5
	$Pt^0$	71.3	4.5
Pt/CeO <sub>2</sub> (DP)	$Pt^{0'}$	70.2	35.3
	Pt <sup>0</sup>	71.4	64.7

The BET surface area of CeO<sub>2</sub> support, Pt/CeO<sub>2</sub> (IR) catalyst, Pt/CeO<sub>2</sub> (IM) catalyst and Pt/CeO<sub>2</sub> (DP) catalyst is 41, 33, 35 and 84 m<sup>2</sup> g<sup>-1</sup>, respectively. It's worth noting that the Pt/CeO<sub>2</sub> (DP) catalyst possesses much higher BET surface area than those of Pt/CeO<sub>2</sub> (IR) and Pt/CeO<sub>2</sub> (IM) catalysts, however, the activity of Pt/CeO<sub>2</sub> (DP) catalyst is poorer than that of Pt/CeO<sub>2</sub> (IR) and Pt/CeO<sub>2</sub> (IM) catalysts. Therefore, the activity of Pt/CeO<sub>2</sub> catalyst is not mainly determined by the BET surface area and there exist other dominative factors for the activity of Pt/CeO<sub>2</sub> catalyst. In

addition, the BET surface area decrease of the Pt/CeO<sub>2</sub> (IM) catalyst and Pt/CeO<sub>2</sub> (IR) catalyst is caused by the addition of Pt species. To reveal the relationship between chemical states of the Pt species and the activity of catalysts, XPS characteristics for fresh Pt/CeO<sub>2</sub> catalyst were carried out, as shown Fig. 6, and the corresponding data analyses are shown in Table 1. The BE values of Pt  $4f_{7/2}$  at 71.1, 72.4 and 74.2 eV are ascribed to  $Pt^0$ ,  $Pt^{2+}$  and  $Pt^{4+}$ , respectively [33]. There are no BE values of Pt  $4f_{7/2}$  attributed to Pt<sup>2+</sup> and Pt<sup>4+</sup> over all Pt/CeO<sub>2</sub> catalysts in Table 1, which suggests that all the Pt species have been reduced into metallic Pt species. For Pt/CeO<sub>2</sub> (IM) catalyst, there are two peaks at 69.6 and 71.1 eV as shown in Fig. 6b. The BE value of Pt  $4f_{7/2}$  at 71.1 eV is ascribed to Pt<sup>0</sup> species, however BE value of Pt  $4f_{7/2}$  at 69.6 eV is far from 71.1 eV, which is defined as Pt<sup>0</sup> species [30, 34]. The negative shift of Pt<sup>0</sup> specie from 71.1 to 69.6 eV is due to the electron transfer from  $CeO_2$ to Pt core [35]. The assignment of Pt species over Pt/CeO<sub>2</sub> (DP) catalyst and Pt/CeO2 (IR) catalyst is similar to that of  $Pt/CeO_2$  (IM) catalyst. The ratios of  $Pt^{0'}$ species to the total



Fig. 7 In situ DRIFTS of the Pt/CeO<sub>2</sub> (IR) catalyst as a function of time at room temperature. a 100 ppm CO/N<sub>2</sub> reaction and b O<sub>2</sub> reaction



Fig. 8 In situ DRIFTS of the Pt/CeO<sub>2</sub> (IM) catalyst as a function of time at room temperature. a 100 ppm CO and N<sub>2</sub> reaction and b O<sub>2</sub> reaction



Fig. 9 In situ DRIFTS of the Pt/CeO<sub>2</sub> (DP) catalyst as a function of time at room temperature. a 100 ppm CO and N<sub>2</sub> reaction and b O<sub>2</sub> reaction



Fig. 10 O2-reoxidaion test over Pt/CeO2 catalysts

Pt species over Pt/CeO<sub>2</sub> (DP) catalyst, Pt/CeO<sub>2</sub> (IM) catalyst and Pt/CeO<sub>2</sub> (IR) catalyst are 35.3, 51.5 and 94.5%, respectively. Therefore the Pt/CeO<sub>2</sub> (IR) catalyst possesses the highest content of Pt<sup>0'</sup>species. It has been proved that the negatively charged Pt<sup>0</sup>'species contributes to O<sub>2</sub> adsorption and chemisorbed oxygen activation [30, 34]. The chemisorbed oxygen should be highly active and deeply involved in the redox cycles of CO oxidation. The charge was further transferred from negatively charged Pt to the chemisorbed oxygen. Meanwhile, the chemisorbed oxygen was activated during the charge transfer. The unique feature due to negatively charged Pt nanoparticles can facilitate the electron transfer and contribute to the activation and transfer of oxygen species. This probably accounts for the high activity for catalytic oxidation of CO at room temperature. In addition, higher electron density on Pt species due to electron transfer from CeO<sub>2</sub> to Pt core can weaken the Pt-CO bonds, and thus enhancing its activity for the CO oxidation [35]. So high content of Pt<sup>0'</sup>specie over Pt/CeO<sub>2</sub> (IR) catalyst should be responsible for the high catalytic activity of the CO oxidation at room temperature. It indicates that the impregnationreduction (IR) method is superior to the impregnation (IM) and deposition-precipitation (DP) methods.

To better study the relationship between catalyst activity and catalyst physicochemical performance, in situ DRIFT spectra of the Pt/CeO<sub>2</sub> catalysts were obtained upon exposure to CO for 50 min, and then upon exposure to O<sub>2</sub> for another 50 min. The peak at 2084 cm<sup>-1</sup> is ascribed to chemisorbed CO over Pt species [23]. Figure 7a shows the in situ DRIFTS spectra of the Pt/CeO<sub>2</sub> (IR) catalyst and the intensity of peak at 2084 cm<sup>-1</sup> increases with the reaction proceeding. It can be observed that the intensity of peak at 2084 cm<sup>-1</sup> seriously decreases in the first 10 min in Fig. 7b, which indicates that most of the chemisorbed CO over Pt species is rapidly oxidized into  $CO_2$ . Then the residual chemisorbed CO is nearly removed in the first 20 min. For Pt/CeO<sub>2</sub> (IM) catalyst in Fig. 8, it takes 50 min to completely remove the chemisorbed CO. However, from Fig. 9, it can be seen that the peaks ascribed to the chemisorbed CO is still observed even after the 50 min reaction. Therefore, the chemisorbed CO can be efficiently removed over the Pt/CeO<sub>2</sub> (IR) catalyst, which is accordance with the results of activity test.

To evaluate the ability of the catalyst to adsorb atmospheric oxygen, an O<sub>2</sub>-reoxidation experiment was carried out. The consumed oxygen peaks were measured by infusing 1.5% O<sub>2</sub> and Ar gas monitored with a TCD at room temperature. O<sub>2</sub>-reoxidation experiment results are shown in Fig. 10. The peak area of Pt/CeO<sub>2</sub> (DP), Pt/CeO<sub>2</sub> (IM) and Pt/CeO<sub>2</sub> (IR) catalyst is 0.041, 0.065 and 0.091, respectively. The peak area of Pt/CeO<sub>2</sub> (IR) catalyst is the biggest, indicating that over Pt/CeO<sub>2</sub> (IR) catalyst more atmospheric oxygen can be adsorbed, which plays an important role for the oxidation reaction [36]. It suggests that high content of Pt<sup>0'</sup>species over Pt/CeO<sub>2</sub> (IR) catalyst can enhance the oxygen adsorption ability, which contributes to the oxidation of CO at room temperature.

# 4 Conclusion

The present research shows that the preparation method significantly affect the activity of  $Pt/CeO_2$  catalyst for the oxidation of CO.  $Pt/CeO_2$  catalyst prepared by the impregnation-reduction method presents excellent activity for the removal of CO at room temperature. Over this catalyst, more  $Pt^{0'}$  species could enhance the adsorption and activation of oxygen species, resulting in the formation of more chemisorbed oxygen ( $O_{II}$ ). The abundant more  $Pt^{0'}$  species are mainly responsible for the high activity of  $Pt/CeO_2$  (IR) catalyst for the removal of CO.

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