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Synthesis of PdO/CeO_2 mixed oxides catalyst for automotive exhaust emissions control

Abdul Hadi, Iskandar I. Yaacob*

Materials Engineering Program, Mechanical Engineering Department, University of Malaya, 50603 Kuala Lumpur, Malaysia

Abstract

PdO/CeO_2 mixed oxide catalysts were synthesized using a modified deposition-precipitation technique. The catalyst was quite effective in oxidizing hydrocarbons (HC) and carbon monoxide (CO), but was not efficient in reducing nitrogen oxides (NO_x). The light-off temperature for CO and HC under stoichiometric conditions was about 130 °C. The presence of CeO_2 enhanced the low temperature activity of the catalyst. About 100% CO conversion was achieved at 235 °C, whereas the conversion for HC reached 77.8% at 245 °C. The promoting effect of CeO_2 also improved the catalyst's ability to operate under lean conditions. The catalyst was able to reduce up to 80% of CO at 250 °C and 66% of HC at 315 °C. The efficiency of the catalyst in reducing NO_x was low and the light-off temperature was not reached over the duration of the experiment. The catalyst started to reduce NO_x only above 240 °C. The highest efficiency of about 27% was achieved at 275 °C.

Keywords: PdO/CeO_2; Low temperature activity; Deposition-precipitation method; Automotive emissions control

1. Introduction

Concern for the danger of automotive toxic gas emissions lead to the development of standards regulating the level of emissions. Emission regulations are applied in European countries since 1988, and more rigorous regulations are planned for USA [1]. In order to meet the required standards, almost every vehicle is today fitted with a catalytic converter. The converter is very effective for reducing emissions, especially for carbon monoxide (CO), hydrocarbons (HC) and nitrogen oxides (NO_x). These emissions are converted to harmless components such as water, CO_2 and N_2. However, the conventional catalytic converter has several disadvantages. First, to establish a high efficiency, the engine operating conditions must be maintained around the stoichiometric point (air/fuel ratio (AFR) of about 14.6 for a petrol engine) [2]. Deviation from this point to either rich or lean conditions severely reduces the effectiveness of the catalytic converter. In practice, the fuel consumption of the engine under stoichiometric condition is high, so that fuel economy is sacrificed. Furthermore, conventional catalytic converters are fully effective only at high temperatures. At low temperatures, the activity of the catalyst is compromised. It is known that more than 60% of the total automotive emission is produced in the first few minutes of operation of the engine (cold start condition) [3,4]. Consequently, pollutants are not completely reduced by the catalyst until the converter reaches the 'conversion' temperature [5]. The temperature at which the catalyst efficiency in reducing emission exceeds 50% is denoted as the 'light-off' temperature and is typically around 350 °C or higher for conventional catalytic converters [6].

The use of Pd-based material supported on CeO_2 for three-way catalysis exhibits promising results in reducing the toxic emissions. There are two major reasons for this renewed interest in palladium. Firstly, palladium is the cheapest noble metal catalyst, so that the use of palladium as three-way catalyst decreases the cost. Also, Pd-based catalysts are more active at lower temperature than other noble metal catalysts, such as Pt- and Rh-based catalysts [7]. It is well known that palladium-based catalysts are very effective in reducing CO and unburned HC emissions around stoichiometric condition [8]. The addition of cerium oxide (CeO_2) enhances the capability of the palladium catalyst. The combination improves the drawbacks of Pd-only catalyst. The oxygen storage component (OSC) function of CeO_2 allows the catalyst to operate over wider air to fuel ratios [9]. Ceria also participates in the water–gas shift reaction and decomposition of nitrogen oxides [10]. The high

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* Corresponding author. Tel.: +60 3 79675204; fax: +60 3 79675317.
E-mail address: iskandar@um.edu.my (I.I. Yaacob).

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activity of Pd/CeO_2 catalyst to reduce CO emission is found to be due to the combined effect of palladium and ceria [11]. Ceria also promotes the palladium and platinum catalyst to exhibit low temperature CO and C_3H_6 oxidation activity and light-off temperature (about 60–70 °C) [12]. The CeO_2 support also enhances the activity of Pd in reducing CO and in CO_2 hydrogenation, particularly for methanol synthesis [13–15].

Although research on the Pd-based catalyst deposited on CeO_2 support is not new, there are several aspects that are still unclear and are of interest to be further investigated. Most of the discussions performed during the last decade are focused on the use of Pd/CeO_2 catalyst for reducing automotive gas emissions at ‘normal’ operating condition. Nearly no study on Pd/CeO_2 catalyst for low temperature activity of CO, HC and NO_x emissions reduction has been reported. Furthermore, most published studies are performed at a laboratory scale using simulated gases. Little information can be found on investigations of Pd/CeO_2 catalysts that are directly tested using actual exhaust gas emissions of automotive engine. There are many conditions in laboratory scale investigation which are principally different from actual engine test investigations. The most important is the effect of engine operating condition on the catalyst efficiency which is impossible to be conducted in laboratory scale investigation. Our research is focused on the activity of the PdO catalyst in reducing toxic gas emissions from a spark ignition petrol engine. PdO catalyst is used instead of Pd metal catalyst because PdO is more active than Pd metal [16–18]. The aim of this research is therefore to develop a PdO deposited on CeO_2 (PdO/CeO_2) catalyst for controlling automotive exhaust emissions. Two aspects of PdO/CeO_2 catalyst performances, i.e. the low temperature activity and the effect of engine operating conditions on the efficiency of the catalyst in reducing toxic gas emissions, are discussed.

2. Experimental

2.1. Preparation procedure and characterizations

The preparation procedure [19] was developed from the deposition-precipitation method which was adopted from the one used by Tsubota et al. [20]. CeO_2 powder (Aldrich) was evacuated at room temperature to eliminate adsorbed water and volatiles from its surface and pores. An aqueous solution of 0.1 M $PdCl_2$ (Aldrich) was added slowly to the evacuated CeO_2. The mixture was then stirred and aged overnight. Pd^{2+} ions were expected to be deposited onto the surfaces and pores of CeO_2. The mixture was filtered. A 0.5 M Na_2CO_3 solution (Riedel) was then added to the unwashed CeO_2 containing adsorbed Pd^{2+} at 70 °C with gentle stirring until the pH of the dispersion reaches 10. The suspension was further stirred for about 1 h. In principle, precipitation of palladium compound occurred exclusively on the support by hydrolysis in the warm solution. The resulting solid was then recovered by filtration and dried for 20 h at 100 °C to remove remaining bound water and volatiles. Finally, the sample was calcined at 500 °C for 5 h to transform the precipitated Pd compound to the desired PdO.

The synthesized catalysts were then characterized using X-ray diffraction (XRD). A Philips expert M’PD X-ray diffractometer employing Cu Kα radiation (wavelength 154.056 pm) was used to investigate the structure and phase of the synthesized catalyst. Spectra were scanned from 20° to 70° (2θ) with a counting time of 1 s per step.

2.2. Catalytic activity study

An engine test bed facility made up of a spark ignition engine fueled with gasoline, a dynamometer, a cooling system and a computer controller was utilized. A 1.5 l Proton Magma 4-cylinder 16-valve spark ignition engine was installed on the test bed and was used throughout the experiments. The engine was operated under steady state conditions. The experiments were performed at constant engine speed of 1500 rev/min with two types of operating condition: (i) constant 15% throttle position for lean condition (AFR > 14.6), and (ii) constant 40% throttle position for stoichiometric condition (AFR $= 14.6$). The engine was fully controlled by a computer with Cadet 6 software. A dynamometer was attached to maintain the variation of load at different speed.

Approximately 5 g of catalyst material (containing 2.03 wt.% Pd) was filled into a glass reactor, supported by a porous silica disc that was fixed at the middle of the reactor. The temperature of the catalyst bed was varied between room temperature and 275 °C using a heating tape that was coiled around the glass reactor. The exhaust gases were sampled at the inlet and outlet of the catalytic reactor. Data were taken after thermal equilibrium was achieved and the concentration of emissions detected by the gas analyzer remained stable. Only three main components of the emissions, i.e. CO, unburned HC, and NO_x were determined.

3. Results

The X-ray diffractogram obtained on PdO/CeO_2 catalyst after calcination at 500 °C is shown in Fig. 1. The pattern is quite similar to that of CeO_2 indicating no obvious phase change occurred in the host of catalyst. The structure is maintained as that of CeO_2, which is face centered cubic (f.c.c.). It is difficult to identify the presence of PdO peaks in the pattern. This is due to the low concentration of palladium oxide deposited in the catalyst system (about 2.03 wt.%). In a narrower scan, a small peak of PdO is detected at 34.20° (2θ), which corresponds to the (1 0 1) peak of PdO. Despite being a ‘weak’ peak, the result proves the presence of PdO in the catalyst system.

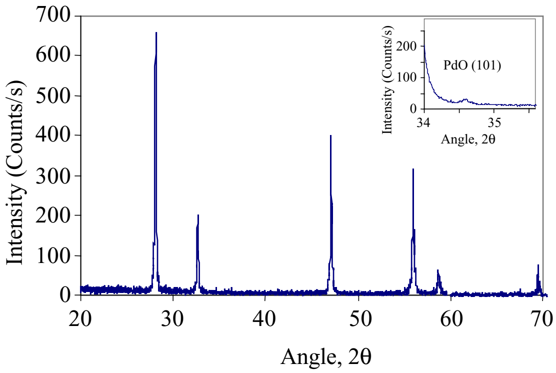

Fig. 1. XRD pattern of PdO/CeO_2 catalyst.

3.1. Regulated emissions

CO emission level at the inlet and outlet of the catalyst bed reactor is shown in Fig. 2. The CO concentration at the start of engine operation is about 1 vol.%. The CO outlet emission is about 0.62 vol.%, indicating that the catalyst is already active. The inlet CO concentrations show a decreasing trend over the range of experiment due to the increase of engine temperature. CO is by-product of incomplete combustion and is essentially partially burned fuel. The formation of CO is higher at the first few minutes of engine operation, specifically during cold start and warm-up operations because complete burning of the fuel is prevented by deficiency in oxygen (rich condition). The formation of CO is low at stoichiometric and lean conditions where complete combustion will most probably occur. The result also shows that the concentration of the outlet CO is sharply reduced over the range of experiment. This is due to the enhancement the catalyst activity at higher temperatures. Almost complete reduction of CO emission is achieved after 40 min.

The concentration of HC at the inlet and outlet of the catalyst bed at increasing experimental duration is shown in Fig. 3. Initially, the inlet HC concentration is about 170 ppm, while the outlet HC concentration is about 85 ppm. Therefore, the light-off temperature (50% efficiency) for HC is achieved. The HC concentration decreases slightly over the range of experiment. Similar to that of CO, the formation of HC is higher at the initial stage of engine operation due to the incomplete combustion during cold start and warm-up operations. The excess HC can also be influenced by the temperature of air/fuel mixture as they enter the engine combustion chamber. Excessively low intake air temperatures can create poor mixing of fuel and air, resulting in partial misfire [21]. The formation of HC is lower at higher temperature because of the complete combustion process. This is consistent with the trend of the curve. However, the result shows that not all HC emission can be reduced over the range of experiment. About 30 ppm of HC emission still remains at the end of experiment probably due to insufficient amount of catalyst used.

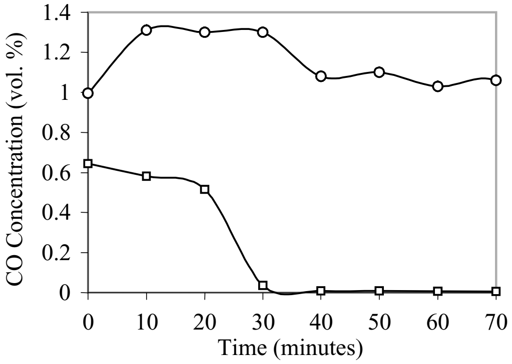

Fig. 2. CO concentration over the duration of experiment. (○: CO inlet; □: CO outlet)

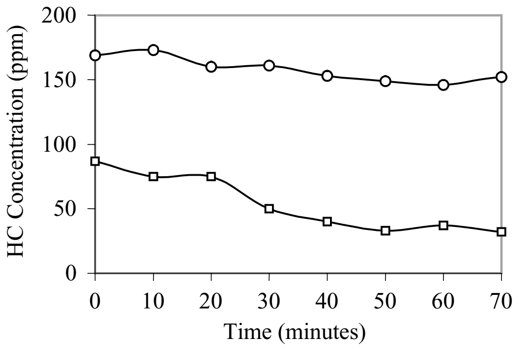

Fig. 3. HC concentration over the duration of experiment. (○: HC inlet; □: HC outlet)

There are two main reasons that cause the increase of the catalyst efficiency in reducing CO and HC emissions at longer engine operating duration. First, the concentration of CO and HC is lower at higher temperature. Hence, the catalyst reduces lesser inlet emission than at lower temperature. Second, the activity of the catalyst becomes higher as its temperature increases.

The inlet and outlet concentration of NO_x is shown in Fig. 4. The result is different from CO and HC. At the initial stage of engine operation, NO_x emission is about 2450 ppm. The outlet concentration is about the same as the inlet, because the temperature required for NO_x conversion is not achieved. The catalyst starts to be active only after

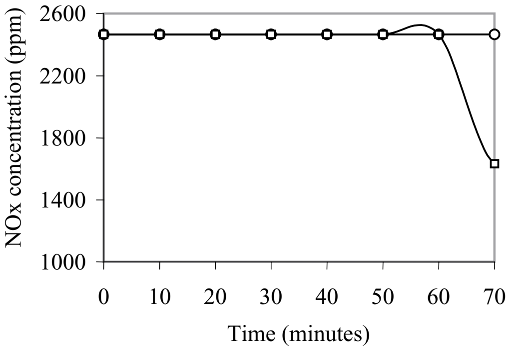

Fig. 4. NO_x concentration over the duration of experiment. (○: NO_x inlet; □: NO_x outlet)

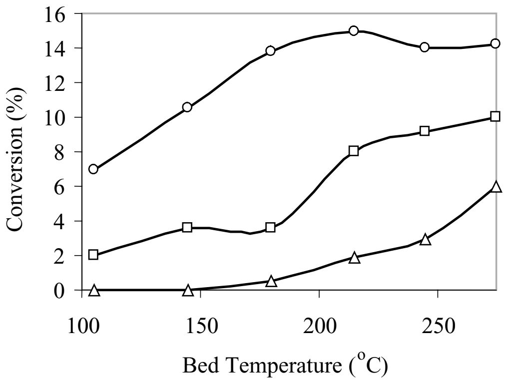

Fig. 5. CO, HC and NO$_x$ efficiencies of pure CeO$_2$. (○: CO; □: HC; △: NO$_x$)

60 min of operation. The palladium is a very good catalyst for CO and HC oxidation, but is not efficient for NO$_x$. The presence of CeO$_2$ in the catalyst system results in the catalyst becoming active after a certain duration of operation because of its high OSC function at higher temperatures [22]. At lower temperatures, CeO$_2$ does not contribute to the NO$_x$ decomposition because the oxygen mobility is too low. CeO$_2$ promotes NO$_x$ decomposition by supplying the oxygen to the reaction.

3.2. Low temperature activity of catalyst

Fig. 5 shows the catalytic activity performance of pure CeO$_2$ in reducing emissions at low temperature. The efficiencies are very low over the temperature range of experiment. Pure CeO$_2$ reduces the emissions by less than 20. Fig. 6 shows the efficiency of the synthesized catalyst in reducing toxic gas emissions as a function of bed temperature. The efficiency increases with increasing temperature. The catalyst becomes active below 100 °C for CO and HC. The highest efficiencies achieved are about 100% for CO at 235 °C and 77.8% for HC at 245 °C. Furthermore, the light-off temperatures of CO and HC are reached below 140 °C. This is significantly lower than conventional catalytic converters. The low temperature activity of the catalyst in reducing toxic emissions is primarily due to the use of palladium material which is known for its ability to reach light-off performances at lower temperatures than other noble metal catalyst. The lower temperature activity of catalyst is also because of the promoting effect of CeO$_2$ in the catalyst system which is caused by reactions initiated on cerium oxide sites that spillover to the Pd-based catalyst [12].

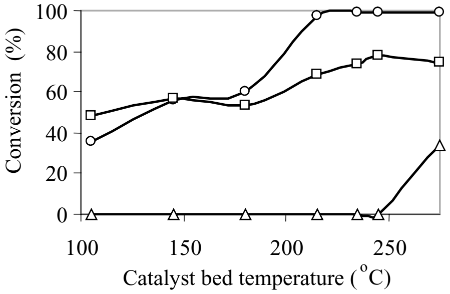

Fig. 6. CO, HC and NO$_x$ efficiencies of catalyst. (○: CO; □: HC; △: NO$_x$)

The efficiency of the synthesized catalyst in reducing NO$_x$ emission is not as significant as for CO and HC emissions. The NO$_x$ conversion efficiency is low and the light-off performance could not be achieved within the temperature range of the experiment. The catalyst starts to be active at above 240 °C. The highest efficiency of about 27% is achieved at 275 °C. The increase of catalyst efficiency in reducing NO$_x$ at high temperature is attributed to the OSC function of CeO$_2$.

3.3. Effect of engine operating condition

Fig. 7 shows the efficiency of the PdO/CeO$_2$ catalyst on lean and stoichiometric conditions. On stoichiometric conditions, the CO light-off performance is reached at about 130 °C and the catalyst is fully effective above 215 °C. On the other hand, the light-off performance on lean conditions is achieved at about 220 °C. The highest efficiency for lean conditions is about 80% which is achieved at about 250 °C. The performance of the catalyst in reducing HC emission shows a similar trend to that of CO. The light-off performance on stoichiometric condition is at about 105 °C and the highest efficiency of about 80% is achieved at 285 °C. Under lean conditions, the light-off performance is reached at 250 °C and the highest efficiency of 66% is achieved at 315 °C.

The catalyst shows better performances in reducing CO and HC emissions for stoichiometric than lean conditions. It is mainly due to the lower CO and HC emission levels produced by the engine. Another reason is the fact that Pd-based catalyst is a good oxidizing catalyst and it is fully active on stoichiometric condition [6]. Furthermore, it is also due to the OSC function of CeO$_2$ that enhances the activity of the catalyst.

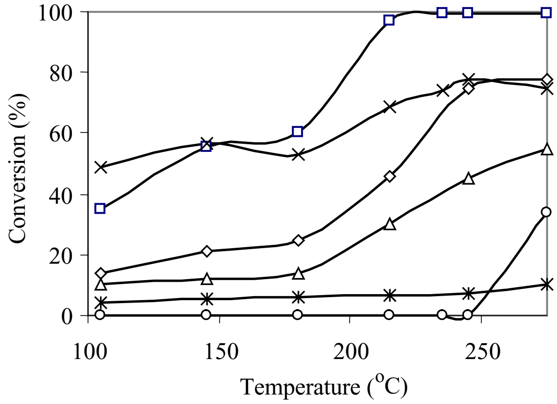

Fig. 7. Catalyst efficiency in reducing CO, HC and NO$_x$ emissions. (□: CO stoichiometric; ◇: CO lean; ×: HC stoichiometric; △: HC lean; ○ NO$_x$ stoichiometric; ✱ : NO$_x$ lean)

Although the catalyst's efficiency in reducing CO and HC emissions on lean conditions is lower than under stoichiometric conditions, our results indicate that the presence of CeO_2 support significantly enhances the ability of PdO catalyst on oxygen deficient conditions.

The efficiency of the synthesized catalyst in reducing NO_x emission on both stoichiometric and lean conditions is very low. However, the catalyst shows better performance on lean condition.

4. Discussion

4.1. Low temperature catalytic activity of ceria

The low temperature activity performances of pure CeO_2 in reducing CO, HC and NO_x emissions from a spark ignition petrol engine indicates that its activity is very low (Fig. 5). Although CeO_2 is able to act as catalyst, it shows unsatisfactory results. CeO_2 is found to be a remarkably efficient catalyst at high temperatures for oxidizing CO emission. Luo et al. [11] demonstrated that CO elimination by pure CeO_2 is strongly dependent on temperature. The material is very effective at temperatures above 250 °C, while the activity at lower temperatures is very low. Similar results are observed by Sazonov and Logvinenko [23] in their study on CO oxidation by rare-earth oxides at 350 °C.

Catalytic combustion of unburned HC and NO_x on CeO_2 at low temperatures is lower than that of CO. Although CeO_2 is supposed to be the most active rare-earth oxide in hydrocarbon oxidation, the catalytic activity of pure CeO_2 is significant at high temperatures only. This is because the participation of oxygen ions of the CeO_2 framework does not occur until 750 °C [24]. For NO_x, CeO_2 alone is not active at all at lower temperatures. The activity of pure CeO_2 in reducing CO, HC and NO_x emissions is mainly due to its OSC function which is caused by oxygen atoms on the surface of CeO_2. It is well known that CeO_2 can easily form oxygen vacancies which can participate in catalytic reactions. This, in turn, implies that oxygen mobility plays an important role in the catalytic properties of CeO_2-based material [25].

4.2. Low temperature activity of catalyst

The activity of PdO/CeO_2 catalysts is much higher than that of pure CeO_2. The higher catalytic activity of the PdO/CeO_2 catalyst in reducing toxic emissions is primarily due to a synergetic interaction between the PdO catalyst and the CeO_2 support. The interaction between the palladium catalyst and the CeO_2 support enhances the lower temperature activity of the catalyst [26,27]. Amongst all noble metals, palladium is the most active at lower temperatures. Our XRD results show palladium to be present as the oxide, which is considered the most active phase of palladium [17]. A mechanism of the promoting effect of CeO_2 to increase the low temperature activity of a noble metal catalyst has been proposed by Torncorna et al. [12] in their study of cobalt oxide and ceria promoted Pt and Pd. The CO oxidation over a Pd/CeO_2 catalyst is initiated by the desorption of CO from the support material at about 100 °C. This leaves free sites for adsorption of oxygen and subsequent reaction between CO and O. When O atoms are present close to the interface between the active catalyst and the support, the reaction may spill over to the catalyst by reaction with CO adsorbed on the catalyst. This is followed by oxygen adsorption on the catalyst and the oxidation reaction. According to this mechanism, the reaction starts on the support material and spills over to the catalyst, where a more rapid light-off can take place. A similar mechanism is anticipated for the oxidation of HC. CeO_2 also retards the transformation of PdO to Pd metal, which is known to be a less active phase.

The activity of catalyst in reducing NO_x at lower temperatures is quite low. The catalyst is active at temperatures above 240 °C only. It is known that palladium-only catalyst shows inefficient NO_x conversion [28–30]. The NO_x reduction on the palladium–CeO_2 system occurs only at high temperatures. The enhanced NO_x activity at high temperature is attributed the high OSC function provided by CeO_2. At low temperatures, the reaction takes place exclusively on the palladium surface. Oxygen storage capacity does not contribute in the reaction, probably because the oxygen mobility is too low.

4.3. Effect of engine operating conditions

The superior performance of palladium-based catalysts in reducing CO and HC emissions from internal combustion engines at stoichiometric condition has been known for a long time [8]. However, under these conditions, palladium catalysts show insufficient NO_x reduction. The catalyst can be improved by addition of a metal oxide support. This combination significantly enhances the NO_x activity of palladium catalyst. In recent years, the challenge in emission control technology was focused on the development of three-way catalysts with high activity at lean operating conditions. Lean burn conditions promise significant improvements in fuel economy, but the problem of NO_x abatement remains to be solved [31].

The result of this study shows that the efficiency of the synthesized catalyst in oxidizing CO and HC emissions at stoichiometric condition is higher than under lean conditions. The better performance of the palladium catalyst at stoichiometric condition is not surprising. However, the oxidation activity of the catalyst decreases at lean conditions as the presence of excess oxygen lowers the gas temperature. Consequently, it reduces the OSC function of CeO_2 by decreasing the oxygen mobility on the surface of the catalyst. However, below 240 °C, the NO_x reduction activity of the catalyst at stoichiometric condition is slightly lower than under lean conditions. The lower inlet NO_x concentration at lean condition is predicted as the main reason for the higher efficiency of the catalyst. At stoichiometric condition, NO_x

production is high due to the high adiabatic temperature. On lean side, NO_x production is reduced because nearly complete combustion dominates. Moreover, NO_x production is reduced because the temperature is decreased due to the presence of the excess oxygen. Reduction of NO_x is enhanced by the presence of CO and HC [7,32]. The NO_x reduction mechanism by CO–NO_x and HC–NO_x interactions is proposed to occur at the active sites which promote the NO_x reduction while the oxidic sites favor the CO or HC oxidations and NO_x decomposition reactions. From our results, the unconverted CO and HC concentrations at lean conditions are higher than stoichiometric condition. Therefore, higher NO_x reduction is achieved at lean conditions. At above 240 °C, the NO_x activity of the catalyst at stoichiometric condition is observed to increase and it is higher than lean conditions. This is possibly due to the effect of OSC function of ceria which is higher at high temperatures.

5. Conclusions

The study of the catalytic activity of a PdO/CeO_2 catalyst for reducing toxic emissions from a spark ignition petrol engine has been carried out. The PdO/CeO_2 catalyst is quite effective in reducing toxic gas emissions, especially CO and HC. The catalyst exhibits lower temperature activity. The catalyst starts to be active in reducing CO and HC at below 100 °C and the light-off temperature is at about 130 °C. The lower temperature activity is due to presence of CeO_2 that enhances the activity of the PdO catalyst at low temperature. The promoting effect of CeO_2 also improves the activity of the PdO catalyst under lean conditions.

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