

Influence of BaO on the catalytic properties and durability of Pd-CZLA for automobile exhaust

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Abstract

Co-precipitation was used to prepare CeO₂-ZrO₂-La₂O₃ (CZL) support, and impregnation was employed to load nano-aluminum sol and Pd to prepare a Pd-CZLA catalyst that can be directly used in car exhaust pipe for the secondary purification of exhaust gas. The specific surface area of the catalyst can reach 143.2 m²/g. At 400 °C, the catalytic efficiency of the catalyst was 76.47%, 70.59%, and 75.41% for C₃H₈, CO, and NO_x, respectively. After modification through doping with 8% BaO in nano-alumina, the catalyst has enhanced high-temperature stability and maintained its high catalytic activity after aging at 1100 °C. Scanning electron micrograph showed that the high-temperature sintering of the catalyst is slowed down when modified by BaO addition. Combined barium oxide and nano-alumina acts as a stabilizer that prevents the crystalline phase transition of alumina at high temperature and further stabilizes its structure. This material also stabilizes Pd dispersion and improves CZA supportive protection at high temperature.

Keywords: Pd-Czla; Catalytic Property; Durability; Barium Oxide

Introduction

Car exhaust is the main source of urban air pollution [1]. The current purification technology for automobile exhaust is to install a three-way catalytic purifier in the exhaust system by using a catalyst that can simultaneously complete the oxidation of CO and hydrocarbons (HCs) and the reduction of NO_x to purify these three harmful substances. The catalyst must exhibit excellent light-off temperature, oxygen storage, and thermal stability [2-4].

CeO₂-ZrO₂-Al₂O₃ (CZA) support has good oxygen storage capacity [5]. Nano Al₂O₃ particles can play a protective role by effectively inhibiting the sintering and phase of cerium-zirconium solid solution [6]. Kagpar et al. pointed out that Al₂O₃ and CeO₂-ZrO₂ modify each other to increase the oxygen storage capacity and improve the dispersion and reduction performance of CeO₂-ZrO₂ composite oxide. CeO₂-ZrO₂-Al₂O₃ carrier is used as a catalyst to improve the purification efficiency of HCs, CO, and NO [7]. CZA is employed to load the honeycomb support and subsequently the active components. The active components directly loaded on CZA support are rarely studied [8-9]. BaO is a

commonly used additive to prevent Al₂O₃ thermal degradation. In addition, Ba can effectively prevent CeO₂ growth due to high temperature fusion [10].

The rare earth metal La is a common three-way catalyst additive [11-13]. Hisaya Kawabata et al. studied ZrO₂ doped lanthanide (La, Ce, Pr, and Nd) single Rh three-way catalysts and found the Rh/La-Zr catalyst has good catalytic performance and anti-hydrothermal aging [14-15]. Yang Yong et al. studied La-Zr modified Pd/Al₂O₃ catalyst for the catalytic oxidation of methane and found that its addition substantially improves the thermal stability of Pd/Al₂O₃ catalyst [16]. Suda et al. prepared three different cerium-zirconium solid solutions and proved that La addition to CeO₂ increases the oxygen storage capacity of the catalyst because in La³⁺ replaces Ce⁴⁺ by providing oxygen vacancies to compensate the positive charge in CeO₂-La₂O₃ solid solution [17].

Compared with Pt and Rh, Pd metal has abundant reserves, low price, and good catalytic activity at low temperature [18], and Pd-based catalysts has good thermal stability and excellent catalytic

activity for HC oxidation [19]. Roy et al. analyzed the catalytic effect of different metal catalysts such as Ru, Rh, Pt, and Pd on NO and proved that Pd catalyst has the highest selectivity and low temperature catalytic activity [20]. In the preparation of a three-way catalyst, Pd has an excellent ability to oxidize and convert HC and CO at low temperatures compared with Pt and Rh catalysts [21, 22].

This paper studies a catalyst that is directly applied to the exhaust pipe of automobiles to perform the secondary purification of automobile exhaust. The catalyst uses cerium–zirconium solid solution as a support, adds an auxiliary agent La, impregnates the nano-aluminum sol, and loads the active component Pd. Its catalytic performance is tested under laboratory conditions. The catalyst was modified with BaO, and its performance and thermal stability are examined.

Experimental

Sample preparation

Preparation of CZLA support

Co-precipitation was used to prepare cerium–zirconium supports with different proportions. The aged samples were obtained by calcining the fresh samples at 1100 °C for 2 h. The cerium–zirconium solid solution was prepared with nine kinds of supports in molar ratios of 1:0, 8:1, 4:1, 2:1, 1:1, 1:2, 1:4, 1:8, and 0:1. The fresh vectors are labeled as CeO₂, CZ81, CZ41, CZ21, CZ11, CZ12, CZ14, CZ18, and ZrO₂. The specific surface area of the cerium–zirconium carrier was measured before and after aging, and the superior carrier was selected.

The cerium–zirconium solid solution was added with different proportions of La, and a fresh cerium–zirconium–lanthanum (CZL) solid solution was prepared through co-precipitation and calcined at 1100 °C for 2 h to obtain aged supports. According to the best cerium–zirconium ratio, five CZL samples with lanthanum nitrate contents of 2, 4, 6, 8, and 10 wt.% were prepared. The catalytic effect of fresh samples and samples after aging was measured, and the optimal loading of La was selected.

The CZL support was prepared according to the optimal ratio screened out in the above steps, and the nano-aluminum sol was impregnated through traditional equal volume impregnation to generate the CeO₂-ZrO₂-La₂O₃-Al₂O₃ (CZLA) support material. The prepared support was ground into powder, and the powder particle size satisfied the 40–60 mesh.

Preparation of Pd-CZLA catalyst

Pd was loaded onto the carrier through dipping. Five catalysts were prepared, and the loadings of Pd(NO₃)•2H₂O were 0.2, 0.4, 0.6, 0.8, and 1.0 wt.%, respectively. The catalytic effect was evaluated, and the best loading of Pd was selected.

Modification of Pd-CZLA catalyst

BaO was added to the nano-alumina for modification, and five

modified aluminum sols with mass fractions of 2%, 4%, 6%, 8%, and 10% were prepared. Five catalysts were also synthesized according to the optimal ratio of catalysts.

Catalyst performance evaluation

Catalyst activity was evaluated in a new plexiglass device from the laboratory. The whole system includes three parts: gas distribution, catalytic reaction and analysis, and testing. The flow chart of the experimental system is shown in Figure 1.

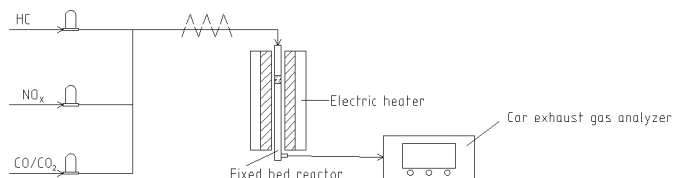


Figure 1: Flow chart of the experimental system

When simulating exhaust gas, the gas flow meters were adjusted as follows: CO to 1.8 L/min, NO_x to 0.8 L/min, and C₃H₈ to 0.8 L/min. The knob of the total flowmeter was rotated to adjust the total flow rate in the range of 3.5–4 L/min. Automobile exhaust gas analyzer was used to measure the content of each component at the outlet entrance. Catalytic activity was examined at 400 °C.

Results and discussion

Ce-Zr ratio

Data on the specific surface area of samples with different proportions of cerium and zirconium before and after aging are shown in Figure 2. The cerium–zirconium solid solution has a superior surface area over CeO₂ and ZrO₂. When the content of added Zr increases, the specific surface area of cerium–zirconium solid solution increases first and then decreases. When the cerium–zirconium ratio is 1: 2, the specific surface area reaches the maximum before and after aging. This phenomenon occurs because using different Ce/Zr molar ratios can give varying structures to cerium–zirconium solid solution. ZrO₂ addition has a good stabilizing effect on Ce³⁺. The presence of Ce³⁺ is conducive to generating additional lattice oxygen defects and improving the material's oxygen storage, cerium atom utilization, and reduction. When the molar ratio of Ce/Zr is suitable, zirconium can maximize the defect space, and the solid solution of cerium–zirconium has good thermal stability. High-temperature aging greatly influences the surface area of the carrier. At high temperature, the cerium–zirconium solid solution is sintered, resulting in a large decrease in specific surface area. The cerium–zirconium solid solution has poor thermal stability, which seriously affects the catalyst activity and thus must be modified.

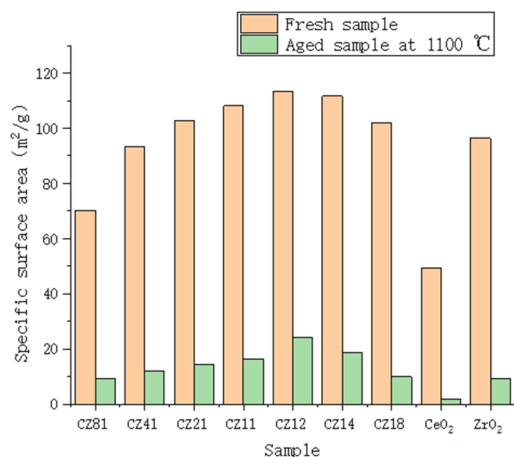


Figure 2: Specific surface area of cerium-zirconium series carrier before and after aging

La-loaded catalyst activity

The catalytic effects of five different lanthanum loading catalysts are shown in Figure 3. When only a small amount of La is added, the catalytic activity and high temperature stability of the sample increase with the increasing La content. This phenomenon occurs because the addition of La causes the lattice distortion of the cerium-zirconium solid solution, thus significantly increasing the specific surface area of the cerium-zirconium solid solution and the mobility of lattice oxygen in the system. When the added amount of La is extremely large, the catalytic activity decreases because the Ce-Zr solid solution particles are filled with La_2O_3 with low specific surface area and low thermal stability, resulting in a significant decrease in the specific surface area before and after aging. This phenomenon affects the catalytic activity of the catalyst. The La-loaded cerium-zirconium solid solution has the best catalytic effect on CH, followed by NOx and CO. The fresh catalyst loaded with 6%La has the best effect on the catalyst efficiency of HC and CO at 65.04% and 52.17%, respectively. The fresh catalyst loaded with 8%La had the best NOx catalytic effect at 68.29% because La addition promotes the reduction of the solid solution of cerium and zirconium, and La^{3+} is introduced into the solid solution of cerium and zirconium to replace parts of Ce^{4+} . Owing to the low valence state, many oxygen vacancies are formed in the crystal lattice, and the oxygen storage performance is improved. Zirconium solid solution absorbs many oxygen molecules and thus is conducive to reducing NOx in the oxygen-depleted state. Meanwhile, HC and CO are easily oxidized in the eutrophic state. After aging at 900 °C, the catalytic activity of the catalyst is not decreased, indicating that the samples calcined at 900 °C have a high stability. After aging at 1100 °C, the catalytic activity of the catalyst is greatly reduced. At high temperature, the cerium-zirconium-lanthanum solid solution is partially sintered, the specific surface area is reduced, the grain size is enlarged, the oxygen vacancy position is reduced, and the reducing ability is reduced. The high temperature of 1100 °C severely damages the surface structure of the catalyst.

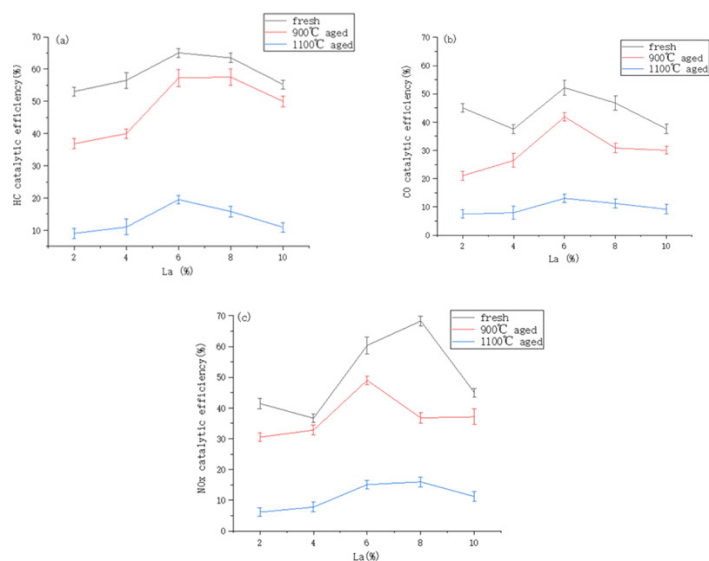


Figure 3: The efficiency of catalysts with different La loading

The catalytic activity of fresh and aged catalysts was comprehensively analyzed, and the cerium-zirconium solid solution loaded with 6%La has the best catalytic performance and hence is selected for comparative study. As shown in Table 1, the specific surface area of the fresh catalyst is the largest. Comparison of the specific surface area revealed that the cerium-zirconium solid solution loaded with 6% La has increased specific surface area. The increase in the specific surface area is beneficial to improving the catalytic efficiency. The specific surface area of the catalyst decreases significantly after deactivation at 900 °C and is reduced to up to 74.63% after deactivation at 1100 °C. The specific surface area of cerium-zirconium solid solution inactivated at 1100°C increases by 8.2 m²/g. Therefore, La addition effectively optimizes the high-temperature stability of cerium-zirconium solid solution.

Table 1: Specific surface area of catalyst loaded 6% La

Sample	fresh	900°C aged	1100°C aged
Specific surface area(m ² /g)	147.8	51.4	37.5

Effect of loading Pd on catalytic efficiency

The catalytic activities of the five catalysts with different Pd contents are shown in Figure 4. The catalytic efficiency of the catalyst added with active component Pd is improved. In particular, the catalytic efficiency after aging at 1100 °C is significantly improved because the precious metal Pd is attached to the surface of the CZLA carrier. Pd has good selectivity, high temperature stability, and high catalytic conversion efficiency for HC. The removal efficiency of the catalyst for three pollutants from automobile exhaust is in the descending order HC, NOx, and CO. When the Pd loading is 0.4%–1.0%, the catalytic efficiency for HC is basically unchanged. When the Pd loading exceeds 0.6%, the catalytic efficiency for NOx decreases. After aging at 900 °C, the catalytic effect is reduced. After aging at 1100 °C, the catalytic effect is lower

than that after 900 °C. Compared with that of CZL catalyst, the effect of high temperature aging at 1100 °C on the catalytic effect is significantly slowed down at 900 °C.

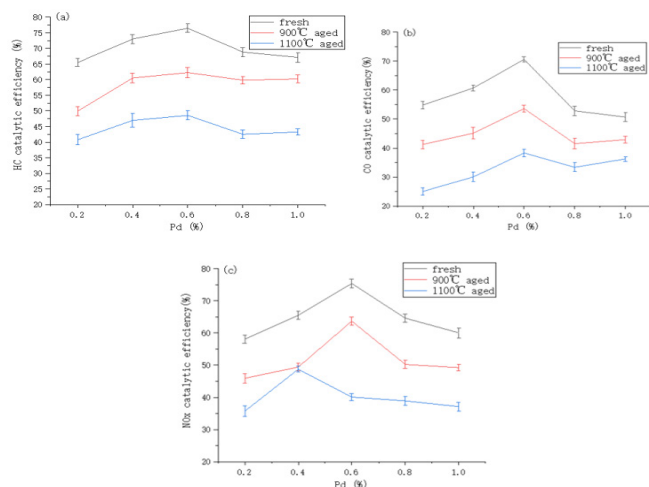


Figure 4: The efficiency of catalysts with different Pd loading

When the Pd loading is 0.6%, the catalyst has the best catalytic efficiency for HC, CO, and NO_x. A catalyst with a Pd loading of 0.6% was selected for further research. Table 2 shows that the specific surface area of the fresh catalyst did not change significantly after impregnating the nano aluminum sol and the active component Pd. The specific surface area after aging at 900 °C is slightly higher than that of CZL catalyst after aging at 900 °C. The specific surface area after aging at 1100 °C is slightly higher than that of CZL catalyst after aging at 1100 °C. This finding indicates that the addition of the nano-aluminum sol coating and active component Pd enhances the high-temperature stability of the catalyst, but the effect is not good. When the temperature is higher than 900 °C, γ -Al₂O₃ transforms into inactive α -Al₂O₃ with a small specific surface area when the temperature increases. This phenomenon affects Pd dispersion on the catalyst surface and weakens the protective effect of nano-alumina on cerium–zirconium solid solution. Thus, modifying the nano-alumina is necessary to improve the stability of the nano-alumina sol coating at high temperature.

Table 2: Specific surface area of catalyst loaded 0.6% Pd

Sample	fresh	900°Caged	1100°Caged
Specific surface area(m ² /g)	144.6	61.4	43.3

Effect of adding BaO on the catalyst

The catalytic efficiency of fresh catalysts with different contents of BaO is shown in Figure 5. The fresh catalysts with different contents of BaO show basically the same catalytic effect. The catalytic efficiency of the fresh catalyst on the three main pollutants of automobile exhaust gas is basically the same as that of the catalyst without BaO addition. Therefore, BaO addition has no effect on the catalytic performance of the fresh catalyst.

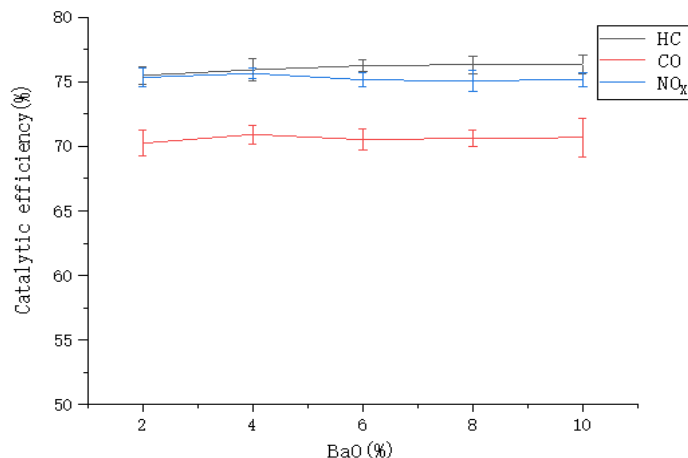


Figure 5: Catalytic efficiency of fresh catalysts with different BaO loadings

Figure 6 shows the XRD pattern of the catalyst. The XRD pattern of the catalyst and the catalyst modified by BaO shows that the four main diffraction peaks in the spectrum are characteristic peaks of the cubic phase structure of fluorite, and the solid solution of this structure has high oxygen storage performance. After BaO modification, neither splitting of diffraction peak nor position change has occurred, indicating that the crystal structure is not changed. The intensity of the diffraction peak of the modified sample is weakened because BaO addition introduces exogenous substances that affect the crystallinity. This finding indicates that BaO addition does not affect the catalytic activity of the fresh catalyst and is consistent with experimental results.

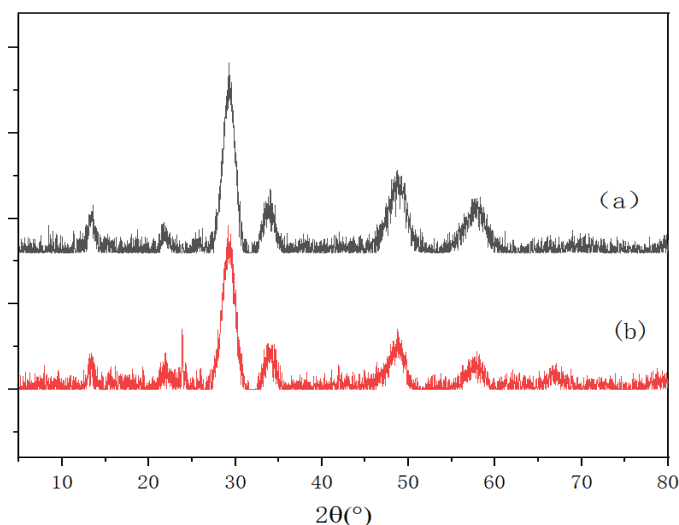


Figure 6: XRD of original and BaO modified samples: Pd/CZAL(a), Pd/CZAL/B(b)

The catalytic effect of catalysts with different proportions of BaO after aging at 1100 °C is shown in Figure 7. Compared with that of the catalyst without BaO, the catalytic efficiency of aging at 1100 °C is improved. The catalyst has the best effect after aging at 1100 °C when the added BaO amount is 8%. The catalytic efficiency of

the catalyst for HC is increased from 48.56% to 62.00%, that for CO is increased from 38.28% to 48.13%, and that for NO_x is increased from 49.09% to 54.26%. The catalysis effect of BaO-added catalyst on HC and CO is enhanced after aging at 1100 °C, but the catalytic effect on NO_x is only slightly improved.

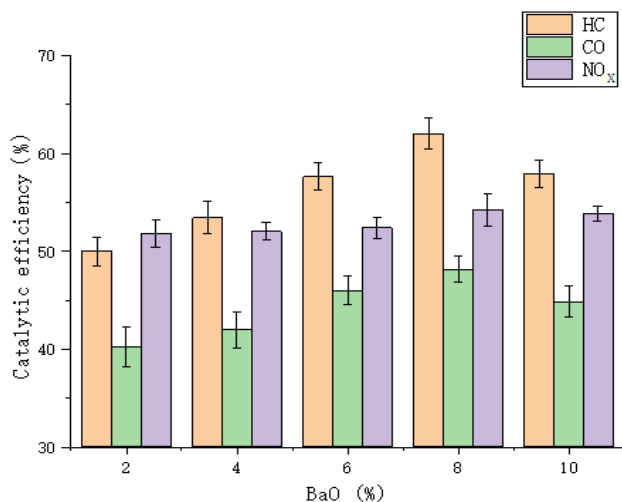


Figure 7: Catalytic efficiency of different BaO supported catalysts after aging at 1100°C

The catalyst added with 8% BaO was selected for research. Table 3 shows the specific surface area of the catalyst after BaO addition. The specific surface areas of the fresh catalyst with BaO and without BaO addition are not significantly different. The specific surface area of BaO-added catalyst after aging at 1100 °C increases from 43.3 m²/g to 54.3 m²/g. This finding fully shows that the BaO-modified catalyst has good high temperature thermal stability. The catalytic activity of the catalyst is reduced at high temperature because this level deactivates the nano-alumina on the surface of the catalyst, thus greatly reducing the specific surface area and porosity of the catalyst, affecting Pd dispersion on the catalyst surface, and shrinking the contact area between the catalyst and gas to reduce the catalyst activity. BaO acts as a stabilizer to prevent the phase transition of γ -Al₂O₃ at high temperature, thus further stabilizing the structure of nano-alumina and maintaining its high surface area. This allows Pd to be well dispersed on the catalyst surface and improve the protection of cerium-zirconium solid solution.

Table 3: Specific surface area of catalyst with 8% BaO added

Sample	fresh	1100°C aged
Specific surface area(m ² /g)	143.2	54.3

Figure 8 is the micrographs of the catalyst surface after the microscope is magnified 3×10⁴ times. Scanning electron micrographs (a), (b), and (c) show that the catalyst is sintered when it was deactivated at 900 °C, but the sintering is not particularly serious. When the catalyst is deactivated at 1100 °C, the sintering becomes serious. The surface structure is critically damaged. Comparison

of (b) and (d) shows that the sintering effect of the catalyst surface after the aging of the catalyst with BaO at 1100 °C is significantly slowed down and is roughly the same as that after the aging of the catalyst without BaO at 900 °C. This finding shows that the modification of nano-aluminum sol by BaO slows the sintering of the catalyst at high temperature.

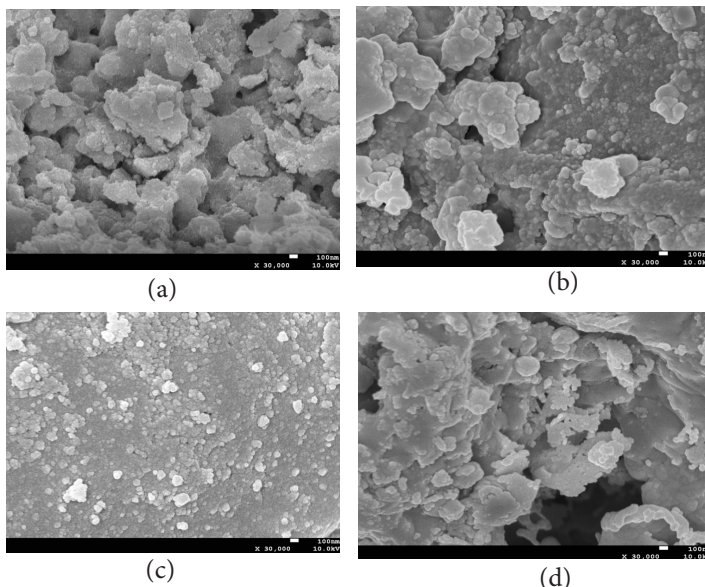


Figure 8: SEMs of different catalysts (a)fresh catalyst (b)900°C aged catalyst (c)1100°C aged catalyst (d) 1100°C aged catalyst with 8% BaO

Conclusion

A new type of catalyst was successfully prepared. This catalyst uses cerium-zirconium-lanthanum solid solution as a carrier and is loaded with nano-alumina and 0.6% of active component Pd through impregnation. The specific surface area of the catalyst can reach 143.2 m²/g. The catalyst can simultaneously purify CH₄, CO, and NO_x at 400 °C, and its catalytic effect reaches 76.47%, 70.59%, and 75.41%, respectively. The catalyst can be applied to the automobile exhaust pipe for the secondary purification of automobile exhaust. The sintering of BaO modified catalyst at high temperature is significantly reduced because the added BaO can act as a stabilizer for nano-alumina, thereby preventing the crystalline phase transition of γ -Al₂O₃ at high temperature and further stabilizing its structure and high surface area. This phenomenon suppresses the agglomeration of crystal grains at high temperature of the cerium-zirconium solid solution while maintaining the dispersion degree of Pd, thus greatly improving the high-temperature stability of the catalyst.

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Conflicts of interest/Competing interests

The authors declare that they have no competing interests

Availability of data and material

Not applicable

Code availability

Not applicable

Authors' contributions

Liqiang QI designed research, Mengmeng LIU and Jingxin LI performed research and analyzed data, Pan ZHANG and Lan CHEN were major contributors in writing the manuscript. All authors read and approved the final manuscript.

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