

Study on supported perovskite-type catalysts for catalytic combustion of volatile organic compounds

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Abstract

$\text{La}_x\text{Ce}_{1-x}\text{M}_y\text{Ni}_{1-y}\text{O}_3$ was supported on $\gamma\text{-Al}_2\text{O}_3$ by equal volume impregnation method. The effects of loading amount, calcination temperature, element type and element ratio in the A-site and B-site on catalytic combustion of toluene were investigated. Then the catalysts were characterized by XRD, BET and SEM. The results showed that the optimum preparation condition of the catalyst was the loading amount of 12%, the calcination temperature of 750°C, and the catalyst form was $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$. Under the action of this catalyst, the ignition temperature T_{50} and complete conversion temperature T_{90} of toluene were 243°C and 303°C, respectively. The supported perovskite catalyst maintained perfect perovskite structure and dispersed uniformly. Its surface area and porosity were greatly increased. The whole structure was fluffy and conducive to practical application.

Keywords: volatile organic compounds, catalytic combustion, perovskite, support

1 Introduction

Volatile organic compounds (VOCs) are the major components in air pollutants, which are organic compounds with the boiling points in the range of 50~260°C and saturated vapor pressure at room temperature for more than 133.3 Pa. Currently there are more than 300 kinds of VOCs having been identified, of which the most common are benzene, toluene, xylene, styrene, trichlorethylene, etc. VOCs have attracted great concern all over the world because of their toxicity to human health and the environment [1]. The main approaches for treatment of VOCs include adsorption [2, 3], membrane separation [4], biodegradation [5, 6], photocatalytic degradation [7], corona method [8] and catalytic combustion technology. Catalytic combustion technology [9] is one of the most effective methods for the elimination of VOCs due to its simple equipment, low energy consumption and high removal efficiency.

Selecting suitable catalysts is the most important thing of the catalytic combustion reaction. There are three types of VOCs removal catalysts, including noble metal catalysts, transition metal oxides and composite metal oxides. Each type has its own advantages and practical limitations. Noble metal catalysts, especially palladium-based catalyst, can exhibit outstanding catalytic behavior at low temperatures. However, they are expensive and easy to be deactivated by poisoning. Non-noble metal catalysts have been more and more attractive due to the lower cost and relatively abundant resources, while they always need a high ignition temperature and the structure is not stable enough [10]. The activity of mixed metal oxides is higher than the single metal oxides due to the interaction of the structure and electronic modulation. Perovskite is one of mixed metal oxides, which is represented by the general formula ABO_3 . The A- and/or B-site of ABO_3 can be substituted by many foreign metal cations without destroying the matrix structure, as long as the tolerance factor is in the range of 0.7~1.1 [11, 12]. The

partial substitution of A and/or B by another metal ions may improve the stability or enhance the activity of the catalyst. In recent years, perovskite catalyst has been widely investigated by researchers because of its advantages of low price, high activity, high temperature stability and good chemical stability. Jiguang Deng et al. [13] removed toluene and ethyl acetate by $\text{LaCoO}_3/\text{SBA-15}$. Hisahiro Einaga et al. [14] investigated the cation A doping of LaMnO_3 for catalytic combustion of toluene, and found that the doped catalyst activity was affected by the calcination temperature. Pecchi et al. [15] prepared $\text{LaFe}_{1-y}\text{Ni}_y\text{O}_3$ by sol-gel method to remove ethanol and ethyl acetate. Zhai et al. [16] prepared LaMnO_3 by co-precipitation method, sol-gel method and spray pyrolysis method, investigating the relationship of different preparation method and the catalytic activity of methane combustion. Most of the studies focus on the element substitution in the A or B cation individually, while little research is about the substitution in both the A and B cations. Therefore, this paper aimed at preparing a series of $\text{La}_x\text{Ce}_{1-x}\text{M}_y\text{Ni}_{1-y}\text{O}_3$ catalysts supported on $\gamma\text{-Al}_2\text{O}_3$ by impregnation method, investigating the influence of preparation conditions on catalytic activity of toluene and emphatically analyzing the effect of the element substitution in the A and B cations.

2 Experimental

2.1 CATALYSTS PREPARATION

The carrier Al_2O_3 was obtained from Aluminum Corporation of Shandong, China with particle diameter of 1~2 mm. First, Al_2O_3 was put into muffle furnace and calcined at 550°C for 4 hours. Specific amount of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{M}(\text{NO}_3)_x \cdot y\text{H}_2\text{O}$ and $\text{N}(\text{NO}_3)_x \cdot y\text{H}_2\text{O}$ were weighed according to molar ratios and dissolved into anhydrous ethanol (M, N were Fe, Co, respectively. When M was Ti, then $\text{Ti}(\text{C}_4\text{H}_9\text{O})_4$ was used.). The solution was stirred vigorously. Then, specific amount

of γ - Al_2O_3 was immersed into the solution by equal volume impregnation method for 12 h, followed by drying at 80°C for 12 h, and finally calcined in a muffle furnace at 750°C for 6 h.

2.2 ACTIVITY TESTS

Catalytic activity tests were conducted in a fixed-bed flow reactor at atmospheric pressure. Schematic representation of the experimental system was shown in Figure 1. It was comprised by the gas generating system and the catalytic combustion system. The concentration of toluene was controlled by adjusting the air flow rate and toluene flow rate. The reactor was a stainless steel tubular reactor with diameter of 20 mm and length of 550 mm, which provided uniform heating of the catalyst bed. Typically, 10 ml of catalyst was packed between the layers of quartz sand. The temperature of catalyst bed was monitored and controlled by temperature controller and the temperature was measured by a type-K thermocouple. The inlet and outlet concentrations of toluene were analyzed by gas chromatograph (VARIAN CP-3800) equipped with FID.

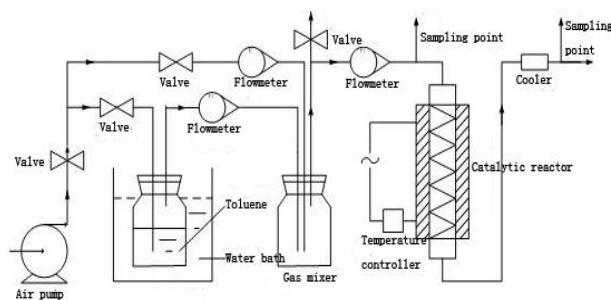


FIGURE 1 Schematic diagram of the experimental catalytic combustion process

2.3 CHARACTERIZATION

The crystal structure of sample was examined by the X-ray powder diffraction (XRD) method, using an X' Pert Pro MPD powder diffractometer made in Netherlands. Measurement of surface area was carried out by the BET nitrogen adsorption method on Micromeritics ASAP2010 automatic adsorption instrument made in America. Scanning electron microscopy (SEM) images were acquired with an S-4800 electron microscope made in Japan to observe surface topography of catalyst.

3 Results and discussion

3.1 CATALYTIC ACTIVITY OF $\text{La}_x\text{Ce}_{1-x}\text{TiO}_3$

3.1.1 Effect of loading amount of active component on catalytic activity

$\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3/\gamma\text{-Al}_2\text{O}_3$ catalyst was prepared in this part. Figure 2 shows the catalytic activities of the five perovskites for toluene oxidation. The specific surface area of the catalyst prepared by conventional method with high calcination temperature is usually less than $5\text{m}^2\cdot\text{g}^{-1}$.

Although the specific surface area of the non-supported catalyst prepared in this study increases to $8\text{m}^2\cdot\text{g}^{-1}$, its activity is still poor. In order to increase the activity, the catalyst must be supported on a carrier so as to reduce the particle size and improve its dispersity. A control test of $\gamma\text{-Al}_2\text{O}_3$ without loading $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3$ is performed under the same conditions. The conversion rate of toluene is very low in the control test. With the increase of temperature, the conversion rate rises slowly, and only reaches to 36% at 410°C . The loading of $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3$ significantly improves the catalytic activity of the catalyst. It is identified from Figure 2 that the activity increases by raising the amount of $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3$ loading. When the loading amount reaches to 12%, the catalyst is the most active. The ignition temperature T_{50} and complete conversion temperature T_{90} of toluene are the lowest. However, when the loading amount is 15%, the catalytic activity decreases. The probably reason is that the active center is not enough at low loading amount, resulting in low activity. Catalytic activity increases with the increase of loading amount. But when loading amount is too high, the active center is covered by metal, resulting in a decreasing activity. Besides, metal is easy to aggregate and lead to some inactivation in reaction of high temperature.

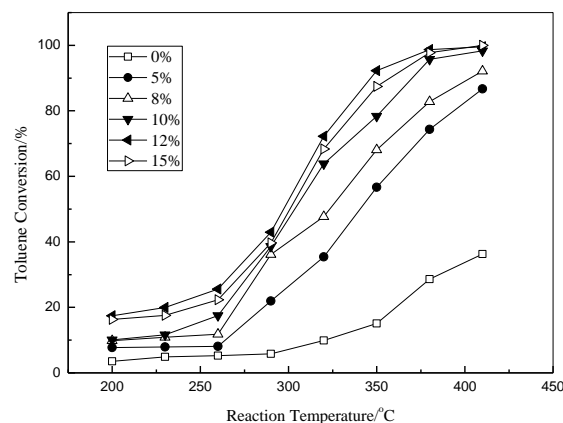


FIGURE 2 Effect of different loading amount of active component on catalytic activity

3.1.2 Impact of calcination temperature on catalytic activity

Figure 3 shows the catalytic activity results obtained for oxidation of toluene over the catalyst $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3/\gamma\text{-Al}_2\text{O}_3$ calcined between 600°C and 1100°C . The catalytic activity shows in general a maximum for the sample calcined at 750°C . With calcination temperature continuing to rise, the conversion rate of toluene under the same reaction temperature reduced gradually at 750°C or more. The calcination temperature has great impact on catalytic activity. Perovskite structure is formed as a single phase upon calcination at $T > 700^\circ\text{C}$. However, as the calcination temperature rises continuously, the grain of $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3$ is sintered, leading to smaller specific surface area and lower activity [17]. T_{90} of toluene over the catalyst calcined at 1100°C is 70°C higher than the one calcined at 750°C , indicating that the thermal stability of the catalyst is not very good.

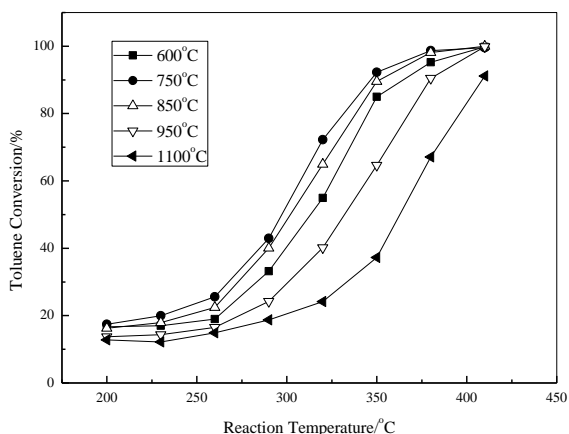


FIGURE 3 Effect of different calcination temperature on catalytic activity

3.1.3 Impact of element ratio of La, Ce on catalytic activity

Figure 4 presents the catalytic activities of the $\text{La}_x\text{Ce}_{1-x}\text{TiO}_3/\gamma\text{-Al}_2\text{O}_3$ ($x=0, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0$) catalysts for toluene oxidation. The substitution of La^{3+} by other rare earth metal ions favors the creation of structural defects, accelerates oxygen diffusion, and induces more surface active oxygen species [18]. Also, it is known that the materials with Ce have high oxygen mobility. $\text{Ce}^{3+}/\text{Ce}^{4+}$ ions cause an exchange of oxygen with the gas phase, resulting in oxygen mobility and therefore higher oxidation activity [19]. Among the catalysts investigated, La-Ce-Ti three-component mixed oxide catalysts show better activity than La-Ti catalyst. When some crystal lattice of the perovskite is substituted by Ce, oxygen vacancies are formed in order to maintain electrical neutrality, and specific surface area of the catalyst changes. The joint action of the oxygen vacancies and specific surface area makes $\text{La}_{0.8}\text{Ce}_{0.2}\text{TiO}_3$ the most active.

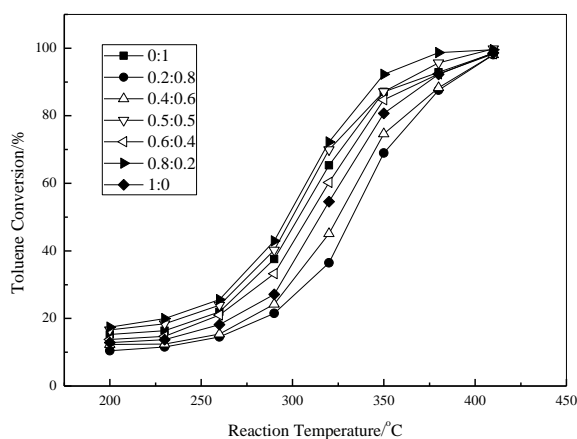


FIGURE 4 Effect of different ratio of La, Ce elements on catalytic activity

3.2 CATALYTIC ACTIVITY OF $\text{La}_{0.8}\text{Ce}_{0.2}\text{Ti}_y\text{M}_{1-y}\text{O}_3$

3.2.1 Impact of element type of M on catalytic activity

The knowledge of structural chemistry shows that the elements in the B cation play a key role on the activity of perovskite catalyst. So $\text{La}_{0.8}\text{Ce}_{0.2}\text{Ti}_{0.8}\text{M}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ ($\text{M}=\text{Fe}, \text{Mn}, \text{Co}$) catalysts were prepared in this part. The catalytic activity of the three samples is plotted in Figure 5. It has

been reported that the catalytic activity of perovskite type $\text{AB}_x\text{B}'_{1-x}\text{O}_3$ is related to the metal-oxygen bond and the free energy of reduction of the cations at B and B' sites [15, 20]. Substitution modifies the surface structure of the catalysts by greatly increasing the oxygen valences in the surface regions, which results in better performance of catalytic activity. Figure 5 shows that the catalyst doped with Mn in the B cation has better activity than the other two samples.

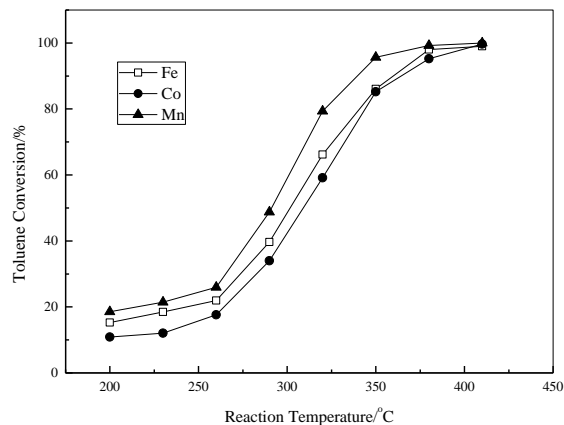


FIGURE 5 Effect of different type of element M in the B cation on catalytic activity

3.2.2 Impact of element ratio of Ti, Mn on catalytic activity

Figure 6 presents the catalytic activities of the $\text{La}_{0.8}\text{Ce}_{0.2}\text{Ti}_y\text{Mn}_{1-y}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ ($y=0, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0$) catalysts for toluene oxidation. The catalytic activity is greatly affected by the M doping amount. When $y=0$, the ignition temperature T50 and the complete conversion T90 of toluene are 257°C and 315°C, respectively, significantly lower than the other six cases. It indicates that $\text{La}_{0.8}\text{Ce}_{0.2}\text{MnO}_3/\gamma\text{-Al}_2\text{O}_3$ exhibits much higher activity than other samples. The possible reason is that Mn has a variety of oxidation states, the modulation between different valences produces lattice defects, thereby improving the catalytic activity [21].

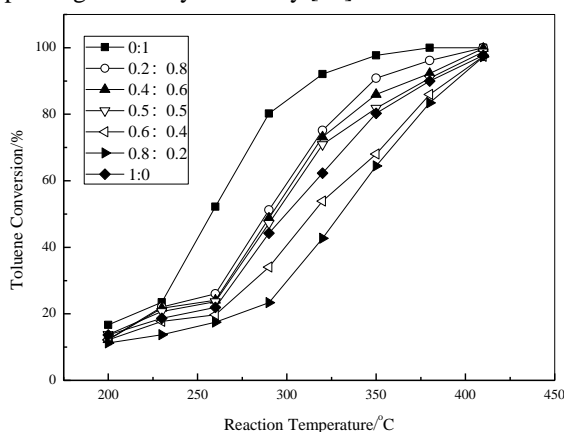


FIGURE 6 Effect of different ratio of Ti, Mn elements on catalytic activity

3.3 CATALYTIC ACTIVITY OF $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_y\text{N}_{1-y}\text{O}_3$

3.3.1 Impact of element type of N on catalytic activity

The studies above show that the substitution with Mn in the

B cation is more effective. So $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{N}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ (N= Fe, Co) catalysts are investigated in the following study. It is known from Figure 7 that the substitution with other transition metal is effective. $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ performs best. T50 and T90 of toluene are 243°C and 303°C over this catalyst. There are different ionic radius and valence state among Fe^{3+} , Co^{2+} and Mn^{3+} . The partial substitution of Mn by Fe and Co results in more lattice defects and oxygen vacancies, which promote the adsorption and desorption of oxygen molecules on the catalyst and improve the catalytic reaction proceeds [22]. As the difference of ionic radius between Mn^{3+} and Co^{2+} is larger, there are more lattice defects in $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$. This is the reason why the activity of $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$ is higher.

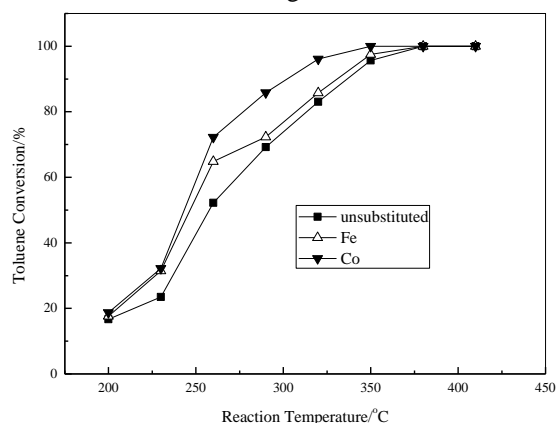


FIGURE 7 Effect of different type of element N in the B cation on catalytic activity

3.3.2 Impact of element ratio of Mn, Co on catalytic activity

The results of catalytic activity of the $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_y\text{N}_{1-y}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ ($y=0, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0$) catalysts in the toluene combustion reaction are shown in Figure 8. The Mn content has a great impact on the activity of the catalyst. When $y=0.8$, the T50 and T90 of toluene are 243°C and 303°C, respectively, significantly lower than the other six cases. With lower Mn content, fewer lattice defects and less space for oxygen conductivity are produced, which leads to low activity. But when Mn content is too high, the larger ion radius of Mn induces larger cell volume and decreasing surface area, also resulting in low activity [23].

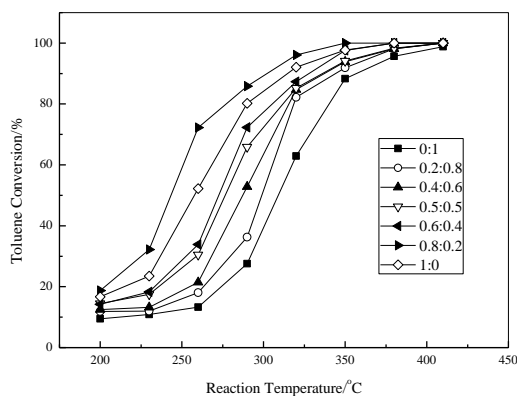


FIGURE 8 Effect of different ratio of Mn, Co elements on catalytic activity

3.4 CATALYST CHARACTERIZATION

3.4.1 XRD characterization

Figure 9 presents the XRD patterns of $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$, $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$, respectively. In Figure 9a, despite the presence of the diffraction lines of $\text{La}(\text{OH})_3$, there are a few strong XRD peaks of perovskite. Figure 9b shows a few observable perovskite diffraction peaks of the investigated catalyst $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$. It is indicated that after being loaded on $\gamma\text{-Al}_2\text{O}_3$, there is little change in the crystal structure of perovskite active group. $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ are well combined, which is more conducive to its practical application.

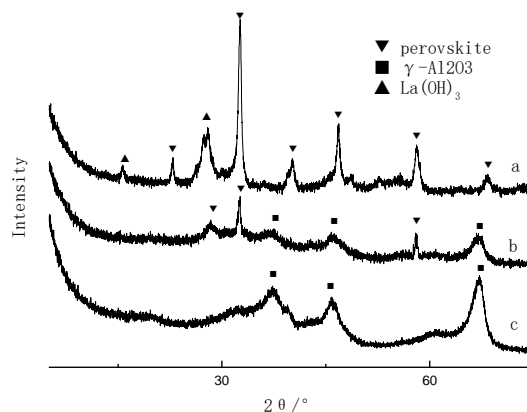


FIGURE 9 XRD Patterns of catalyst samples: a) $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$; b) $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$; c) $\gamma\text{-Al}_2\text{O}_3$

3.4.2 BET characterization

The BET analysis data of these samples are summarized in Table 1. The specific surface area of $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$ is only $8.0375 \text{ m}^2 \cdot \text{g}^{-1}$, while increasing to $116.0388 \text{ m}^2 \cdot \text{g}^{-1}$ after being loaded on $\gamma\text{-Al}_2\text{O}_3$. Although some of the pores are blocked after loading, perovskite can be dispersed more uniformly, thereby increasing the number of active centers. There is also a large variation in the pore size of the catalyst before and after loading. The pore size decreases from 19 nm to 11 nm. The decreasing of pore size produces more active points, which are more conducive to the improvement of activity.

TABLE 1 S_{BET} of catalysts

Catalysts	Surface area / $\text{m}^2 \cdot \text{g}^{-1}$	Pore volume / $\text{cm}^3 \cdot \text{g}^{-1}$	Pore size /nm
$\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$	8.0375	0.0387	19.2599
$\gamma\text{-Al}_2\text{O}_3$	170.2937	0.4586	10.7717
$\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3/\gamma\text{-Al}_2\text{O}_3$	116.0388	0.3280	11.3074

3.4.3 SEM characterization

Figure 10 shows the SEM images of three samples. As can be seen in the figure, $\gamma\text{-Al}_2\text{O}_3$ shows puffy porous and loose structure, while the structure of $\text{La}_{0.8}\text{Sr}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$ is dense and flaky. The supported $\text{La}_{0.8}\text{Ce}_{0.2}\text{Mn}_{0.8}\text{Co}_{0.2}\text{O}_3$ is

dispersed more uniformly by γ -Al₂O₃. The sample is fluffy and the fine pores increase significantly, which is in good agreement with BET analysis data. It is indicated that perovskite and γ -Al₂O₃ are organically combined, enhancing the activity of catalyst.

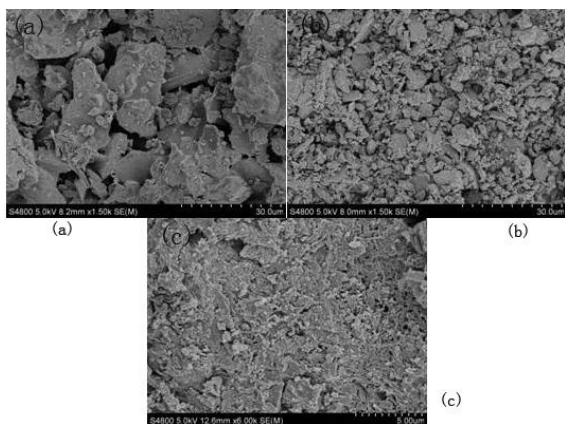


FIGURE 10 SEM images of catalyst samples (a) γ -Al₂O₃; (b) La_{0.8}Ce_{0.2}Mn_{0.8}Co_{0.2}O₃/ γ -Al₂O₃; (c) La_{0.8}Ce_{0.2}Mn_{0.8}Co_{0.2}O₃

4 Conclusions

The effects of preparation conditions of supported perovskite catalysts were emphatically investigated. A series of La_xCe_{1-x}M_yN_{1-y}O₃/ γ -Al₂O₃ catalysts were prepared by equal volume impregnation method. The catalytic activity for combustion reaction of toluene was investigated of these samples. La_{0.8}Ce_{0.2}Mn_{0.8}Co_{0.2}O₃/ γ -Al₂O₃ with loading amount of 12% and calcination temperature of 750 °C performed best. The element substitution in the A and B cation of perovskite changed the oxidation state of the metal ions, which leading to the improvement of catalytic activity. There was little change in the crystal structure of perovskite after loading. Perovskite was dispersed uniformly over the surface of γ -Al₂O₃ and organically combined with it.

Acknowledgments

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