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Effect of surface area of La–K–Mn–O perovskite catalysts on diesel particulate oxidation

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ABSTRACT

Catalytic combustion of diesel particulate matter (PM) was carried out over K-containing perovskite-type oxide of $La_{0.8}K_{0.2}MnO_3$ (LKM82) with different specific surface areas. The surface area of LKM82 was controlled by changing the preparation methods and conditions. PM combustion temperature evaluated from thermogravimetric analysis decreased by 150 °C and 200 °C under tight-contact and loose-contact conditions, respectively, with an increase in catalyst surface area from 1.1 $m^2 g^{-1}$ to 18.5 $m^2 g^{-1}$. Ceramic foam filters coated with LKM82 catalyst were fabricated to further investigate the PM combustion performance under the practical contact condition between the catalyst and PM. The PM combustion performance was also improved with the increase in the surface area of the catalyst coated on the ceramic foam filter. These findings strongly suggest that surface area is one of the important factors controlling catalytic performance for diesel particulate combustion.

1. Introduction

Diesel engines have advantages over gasoline engines in reducing CO_2 emission and fuel consumption. However, diesel particulate matter (PM) and nitrogen oxides (NOx) in their exhaust gases cause serious environmental and health problems. Therefore, the development of catalytic systems for the removal of PM and NOx is highly required. Catalyst coating on a diesel particulate filter (DPF) is one of the effective methods to control PM emission. We have reported that potassium–manganese-based oxide catalysts such as $La_{1-x}K_xMnO_3$ perovskites and $K_2Mn_4O_8$ show high performance for simultaneous removal of PM and NOx under the condition where catalyst is tightly contacted with PM [1]. It has been reported, however, that the PM combustion performance strongly depends on the contact conditions between PM and catalysts. Metal oxides such as Co_3O_4, V_2O_5 and Fe_2O_3 showed high performance for PM combustion when these catalysts and PM were tightly contacted, whereas the combustion performance deteriorated under “loose-contact” condition [2,3]. Since PM trapped in practical diesel exhaust gases is loosely contacted with catalysts on a DPF, our research target has been focused on the improvement of catalytic performance for PM removal under loose-contact conditions.

In the last decades, various types of catalysts have been used for the PM combustion under loose-contact conditions. Metal salts and mixed metal salts such as Cu/K/Mo/Cl, Cu/K/V, Cu/K/V/Cl, $K_{0.7}Cu_{0.3}VO_3$ + KCl, V_2O_5 + K_2SO_4 showed catalytic performance for the reaction [3–7]. In these catalysts, their high performance under “loose-contact” condition was ascribed to the low melting temperatures of the catalyst materials, which results in the increase of the contact points between the active species of the catalysts and PM. However, these catalysts lack thermal stability under the practical operating condition and are not suitable for practical use. On the other hand, mixed metal oxides are thermally more stable than the metal salts, and some authors have reported the catalytic activity of mixed metal oxides for PM combustion under loose-contact conditions. Wang et al. have reported that nano-sized $La_{1-x}K_xMnO_3$ is highly active under loose-contact condition, because the nano-sized catalyst particles can contact well with PM under loose-contact condition [8]. However, the factors affecting PM combustion performance under the loose-contact conditions have not been systematically investigated.

In this paper, we report the catalytic performance of perovskite-type oxides of $La_{0.8}K_{0.2}MnO_3$ (LKM82) for PM combustion by using two kinds of reaction systems: the one used powder catalysts for the combustion of carbon black, a model substance of PM, and the other employed the catalyst coated on ceramic foam filters for the treatment of practical diesel exhaust gases. The present study revealed that the surface area of the catalyst is one of the important factors controlling the PM combustion performance. These

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findings gave us important information on obtaining high-performance for catalytic PM combustion.

2. Experimental

2.1. Catalyst preparation

Perovskite-type $La_{0.8}K_{0.2}MnO_3$ (LKM82) oxides were prepared by the malic acid-aided process [9,10], and the conventional evaporation-to-dryness process.

2.1.1. Malic acid-aided process

To the mixed aqueous solution dissolving $La(NO_3)_3{\cdot}6H_2O$, KNO_3, and $Mn(NO_3)_2{\cdot}6H_2O$ in the molar ratio 0.8:0.2:1.0, malic acid was added so that the molar ratio of malic acid to the total metal cations was 1.5–1.0. The mixed aqueous solution was evaporated to dryness with stirring, followed by heating at 350 °C for 2 h on a hot plate and calcination at 550 °C, 600 °C, and 750 °C for 5 h in air.

2.1.2. Conventional evaporation-to-dryness process

$La(NO_3)_3{\cdot}6H_2O$, CH_3COOK and $Mn(CH_3COO)_2{\cdot}4H_2O$ were dissolved into distilled water in the molar ratio 0.8:0.2:1.0, and the mixed aqueous solution was evaporated to dryness with stirring. The residue was heated at 350 °C for 2 h to decompose the metal salts, followed by calcination at a temperature in the range from 850 °C to 1050 °C for 5 h in air.

The crystal structure and specific surface area of the catalysts were measured by XRD using Cu Kα radiation (Rigaku, RINT2200) and N_2 adsorption at 77 K (BET method), respectively.

2.2. Catalyst coating on ceramic foam filters

In this experiment, ceramic foam filters (CFF; 38 mm diameter, 50 mm length) were used due to the high PM trap efficiency, high durability to thermal shock, and good contact throughout the filter between PM and catalysts by the deep filtration mechanism [12–15]. Porous ceramic foam filters made of α-Al_2O_3 were obtained from Kurosakiharima Corporation and used for the preparation of the catalyst-coated CFF (cat-CFF). The average diameter of the pore was 2.0 mm. The catalyst was coated on the CFF by dip coating method (Fig. 1). A CFF was dipped into the aqueous solution containing appropriate amounts of $La(NO_3)_2{\cdot}6H_2O$, CH_3COOK and $Mn(CH_3COO)_2{\cdot}4H_2O$ under reducing pressure for 30 min. The CFF was lifted up from the solution and extra solution remaining in the pore of the CFF was blown off by an air gun, followed by drying and precalcination at 350 °C in air. This operation was repeated until the weight of the catalyst loading reached the targeted value, and then the CFF was calcined at a temperature between 700 °C and 1000 °C for 10 h. The crystal structure of cat-CFFs and surface morphology was examined by XRD (Rigaku, RINT2200) and SEM (HITACHI, S-3000N), respectively. Specific surface area of cat-CFFs was measured by Kr adsorption at 77 K by BET method.

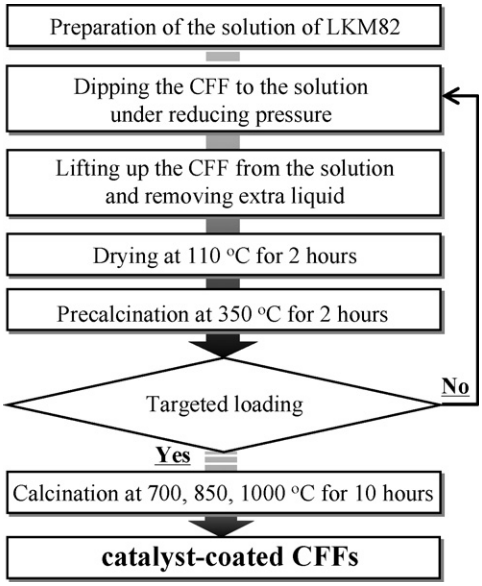


Fig. 1. Preparation of catalyst-coated ceramic foam filter (cat-CFF) by dip-coating method.

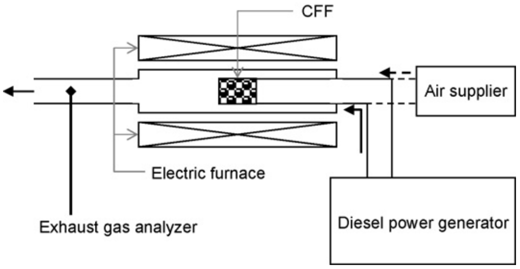


Fig. 2. Experimental set-up of the catalytic reaction system for the evaluation of PM combustion performance of catalyst-coated ceramic form filters.

2.3. PM combustion performance

PM combustion performance of powder catalysts was evaluated by thermogravimetry (TG). Carbon black (CB, Sigma–Aldrich corp.) was used as a model substance of PM. The catalyst and CB (*ca.* 5 wt%) were mixed under the two kinds of contact modes; tight-contact (TC) and loose-contact (LC) modes. Under the TC mode, the catalyst and CB was well mixed in an agate mortar for 10 min. Under the LC mode, on the other hand, the catalyst and CB were mixed by spatula carefully for 10 min, and the contact condition is reportedly similar to that between PM and a catalyst under the condition of practical exhaust gas treatment [11]. These mixtures were heated from 120 °C to 800 °C at a rate of 10 °C min^{-1} in a flow of synthetic air (21% O_2 and 79% N_2, 100 mL min^{-1}). The temperature of T_{max} at which the rate of CB combustion became largest, was evaluated from TG curves and used as an index of PM combustion temperature.

PM combustion performance of the cat-CFF was evaluated from temperature programmed oxidation (TPO) measurements under the practical contact condition between the catalyst and PM. Fig. 2 shows the experimental set-up of the catalytic reaction system for the evaluation of PM combustion performance of cat-CFF. The exhaust gas from the diesel power generator (YANMAR Co., Ltd., YDG500S) was used as the practical exhaust gas. The cat-CFF was fixed in the exhaust gas line and exposed to the exhaust gas for 120 min to trap PM on the cat-CFF (SV = 680,000 h^{-1}). Subsequently, the cat-CFF carrying trapped PM was heated to 800 °C at a rate of 10 °C min^{-1} in an air flow (2 L min^{-1}). The concentration of CO_2 and CO in the effluent gas was determined by TESTO 350XL (TESTO INC.). The performance for PM combustion of the cat-CFFs was evaluated by the temperature (T_C) in the TPO measurement at which the CO_2 concentration became maximal.

3. Results and discussion

3.1. PM combustion over LKM82 powder catalysts under tight- and loose-contact conditions

Table 1 summarizes the preparation conditions and the specific surface areas of LKM82 catalysts. Here, calcination temperature

Table 1
Specific surface area of the catalyst prepared by several conditions

Preparation method	Starting materials					Calcination temperature (°C)	Specific surface area (Sa) ($m^2\ g^{-1}$)
	La	K	Mn	Organic acids	Molar ratio of organic acid to total metal cations		
(1)	Nitrate	Nitrate	Nitrate	Malic acid ($C_4H_6O_5$)	1.5/1.0	550	18.5
						600	16.7
						750	14.7
(2)	Nitrate	Acetate	Acetate	–	–	850	8.2
						950	3.1
						1050	1.1

and starting materials were variously changed. In the malic acid-aided process, metal nitrates and a malic acid were used as raw materials and a complexing agent, respectively, and the calcination temperature was changed from 550 °C to 750 °C. The surface area monotonically increased from 1.1 $m^2\ g^{-1}$ to 18.5 $m^2\ g^{-1}$ with a decrease in the calcination temperature irrespective of the preparation methods.

Fig. 3 shows the XRD patterns of LKM82 catalysts prepared by the malic acid-aided and evaporation-to-dryness processes. The characteristic peaks of perovskite-type oxides were observed for all the catalyst samples. $K_2Mn_4O_8$ was formed as the byproduct when the catalysts were calcined at lower temperatures (550 °C and 600 °C). On the other hand, no other peaks were observed for the samples when they were calcined at a temperature of 750 °C or above, indicating the formation of single-phase LKM82 perovskite oxides. The temperatures of the by-production of $K_2Mn_4O_8$ were lower than those reported earlier (800 °C and 850 °C) [1,8,16]. The LKM82 catalysts calcined at 550–850 °C were suggested to have a 'pseudo-cubic unit cell', which is slightly distorted from the cubic structure [1]. When the calcination temperature increased to 950 °C and 1050 °C, peak splitting was observed and the patterns were indexed on the basis of a hexagonal unit cell. There are two possible reasons to explain the change of XRD patters. As recognized from Table 1, specific surface area of LKM82 decreased steeply over the calcination temperature of 950 °C, indicating the occurrence of the growth of crystalline and the increase in the crystallinity. The LKM82 catalysts synthesized below 850 °C had higher specific surface area and therefore smaller particle size and lower crystallinity. In this case, XRD peaks become broader. Even when the structure is hexagonal, two neighboring peaks that should be separated are merged into one peak and the XRD pattern looks cubic (pseudo-cubic). The other possibility is the real structural change caused, for example, by volatilization of K. At the present, we cannot exclude the possibility that structural and compositional changes affect the catalytic performance, and it will be clarified in future studies.

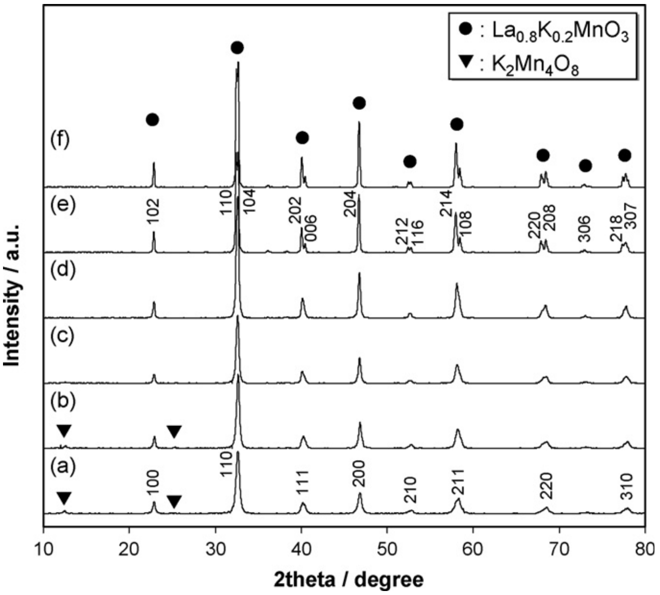


Fig. 3. XRD patterns of $La_{0.8}K_{0.2}MnO_3$ prepared by (a–c) malic acid-aided and (d–f) evaporation-to-dryness processes. Calcination temperature: (a) 550 °C, (b) 600 °C, (c) 750 °C, (d) 850 °C, (e) 950 °C, and (f) 1050 °C. Indices in (a) and (e) are based on cubic and hexagonal unit cells, respectively.

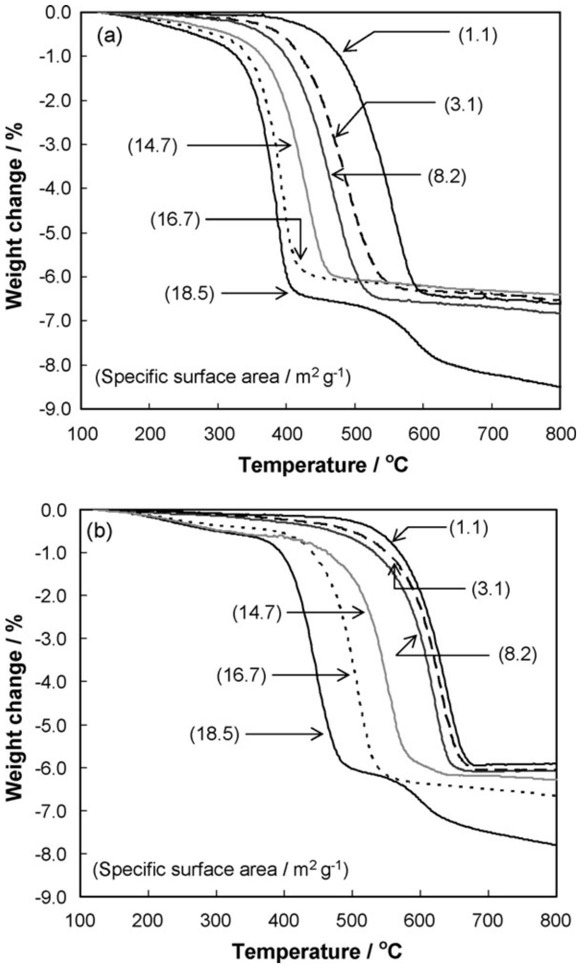


Fig. 4. TG curves of mixture of carbon black (5 wt%) and $La_{0.8}K_{0.2}MnO_3$ with different surface areas under (a) tight- and (b) loose-contact conditions.

Table 2
Specific surface area (Sa) and T_{max}[a] of the $La_{0.8}K_{0.2}MnO_3$ catalysts under TC and LC modes

Preparation method[b]	Calcination temperature (°C)	Specific surface area (Sa) ($m^2\,g^{-1}$)	T_{max}[a] (°C)	
			Tight contact ($T_{max,TC}$)	Loose contact ($T_{max,LC}$)
(1)	550	18.5	384	439
	600	16.7	396	503
	750	14.7	415	546
(2)	850	8.2	457	618
	950	3.1	485	628
	1050	1.1	535	638

[a] The temperature at which the rate of CB combustion becomes largest on the TG curve.
[b] See Table 1.

Fig. 4 shows the TG curves of CB combustion using the catalysts with different surface areas under both TC and LC modes. In general, the weight of 5 wt% CB/LKM82 mixtures decreased steeply due to the CB combustion with increasing temperature and reached constant values after the exhaustion of CB. It is worth to notice that we have investigated the products in gas phase during CB combustion reaction by TCD gas chromatograph (Shimadzu, GC-8A) in a separate experiment, and confirmed that CO_2 and CO was mainly formed and was never detected, respectively. Further weight losses were observed above *ca.* 550 °C for the catalyst with the largest surface area (18.5 $m^2\,g^{-1}$). Since the calcination temperature of the catalyst was 550 °C, the weight losses are possibly ascribable to the decomposition of some residual species such as carbonates formed in the preparation process. The preservation of the perovskite structure after the CB combustion experiments up to 800 °C was confirmed by XRD for all the samples.

The ignition temperature of CB combustion at which CB started to combust ranged between 300 °C and 400 °C under the TC mode and between 370 °C and 540 °C under the LC mode, indicating that CB tightly contacted with the catalyst is ignited more easily than that loosely contacted. The TG curve shifted to the lower temperature side with increasing the catalyst surface area under both contact modes. This clearly shows that the PM combustion performance is improved with the increase in catalyst surface area.

From TG curves, the values of T_{max} giving the highest rate of weight loss due to the CB combustion were obtained and they are summarized in Table 2. The T_{max} value monotonically decreased with increasing the catalyst surface area from 1.1 $m^2\,g^{-1}$ to 18.5 $m^2\,g^{-1}$ under the TC mode. Under the LC mode, on the other hand, T_{max} was almost unchanged from 1.1 $m^2\,g^{-1}$ to 8.2 $m^2\,g^{-1}$ and was close to the temperature at which CB combusted on α-Al_2O_3 (*ca.* 600 °C). The value greatly decreased when the catalyst surface area exceeded 8.2 $m^2\,g^{-1}$. Thus, the promotion effect of catalyst for PM combustion was significant under high surface-area conditions. It is noteworthy that the decrement of T_{max} by changing surface area from 1.1 $m^2\,g^{-1}$ to 18.5 $m^2\,g^{-1}$ was *ca.* 150 °C and 200 °C under TC and LC modes, respectively. This strongly indicates that the catalyst surface area greatly affects the PM combustion performance under both TC and LC modes and that the promotion effect by the increase in surface area is more prominent in the LC mode.

As described above, XRD patterns suggested the possibility of the structural change between 850 °C and 950 °C. However, no clean gap of the catalytic performance was observed between catalysts calcined at 850 °C and 950 °C. This indicates that the effect of the surface area is more important and that it dominates over the effect of structural change even when the structural change takes place.

3.2. PM combustion over CFF coated with LKM82 catalyst under practical contact condition

As described above, LKM82 catalysts with high surface area is effective for PM combustion especially under the LC mode. Therefore, LKM82 catalyst was coated on the CFF to investigate their performance for PM combustion under the practical contact condition. Fig. 5 shows the XRD patterns of the CFFs on which LKM82 catalyst was coated (denoted by cat-CFF) by dip-coating method under various conditions (Table 3). The peaks due to the perovskite phase appeared after the catalyst was coated and calcined in the temperature range 700–1000 °C in addition to the strong peaks of α-Al_2O_3 which was the base component of the CFF. No other peaks were detected. Thus, the perovskite catalysts were conveniently prepared on CFFs by using the dip-coating method. Table 3 listed the weight of LKM82 catalyst coated on the CFF. The weight of the coated catalysts was in the range of 2.58–3.17 wt% when the dipping condition was the same and the calcination temperature was different (Methods B–D). This indicates that the reproducibility of the catalyst loading is relatively good in spite of the quite simple procedure.

Fig. 6 shows SEM images of the surface of the CFFs with and without the catalyst coating. The pores with the size of approximately 1–10 μm were observed on the bare surface of CFF (Fig. 6(a)). The pores disappeared and the surface morphology

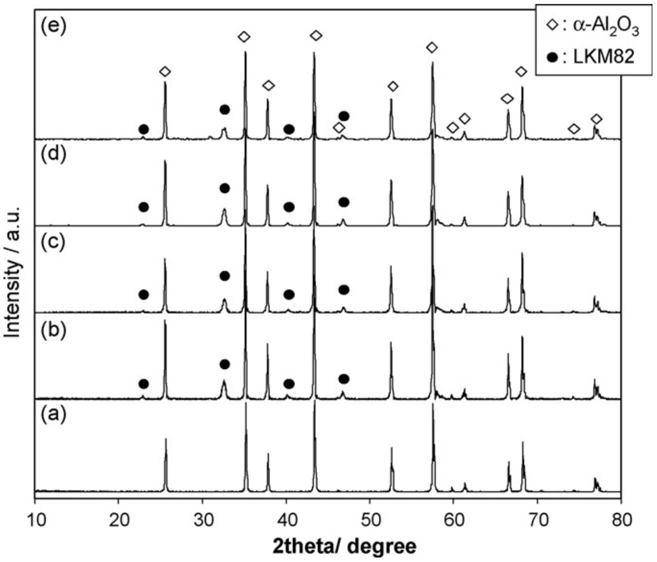


Fig. 5. XRD patterns of (a) bare ceramic foam filter and $La_{0.8}K_{0.2}MnO_3$-coated ceramic foam filters prepared by (b) Mathod A, (c) Method B, (d) Method C and (e) Method D. See Table 3 for the details of the preparation method.

Table 3
Preparation condition and surface area data of catalyst-coated ceramic foam filters

Preparation method	Concentration of LKM82 solution (mol/L)	Times of dipping (times)	Calcination temperature (°C)	W[a] (wt%)	Sa′[b] ($m^2\ g_{\text{-catCAF}}^{-1}$)	Sa″[c] ($m^2\ g_{\text{-cat}}^{-1}$)
A	0.3	3	850	3.52	0.34	9.5
B	1.0	1	700	3.17	0.42	13.4
C	1.0	1	850	2.58	0.26	10.0
D	1.0	1	1000	2.98	0.16	5.2

[a] Weight ratio of the loaded catalyst to the cat-CFF.
[b] Specific surface area of cat-CFFs.
[c] Specific surface area of the coated catalyst on CFFs.

was completely changed after the catalysts were coated by Methods A–D (Fig. 6(b–e)), implying that the whole surface of the CFF was covered with the LKM82 catalyst by the simple dipping method.

Table 3 shows the surface area of cat-CFFs (denoted by Sa′ [$m^2\ g_{\text{-catCFF}}^{-1}$]) and that of the coated catalysts on the CFFs (Sa″ [$m^2\ g_{\text{-cat}}^{-1}$]), which was calculated by Eq. (1):

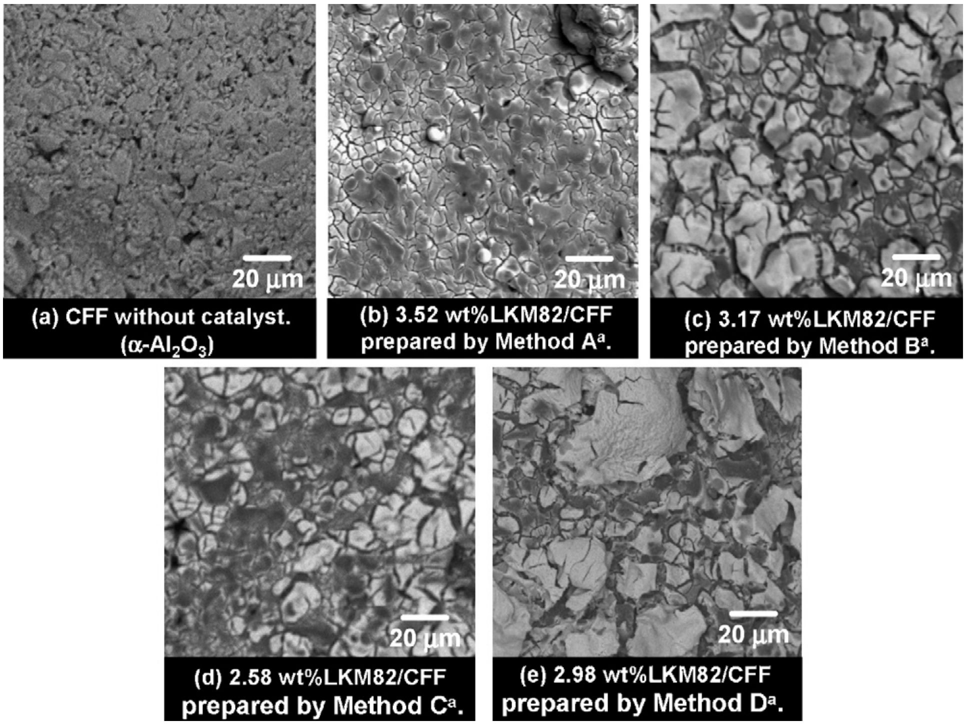
$$\mathrm{S\,a''}[\mathrm{m}^2\ \mathrm{g}_{-\mathrm{cat}}{}^{-1}] = \frac{\mathrm{S\,a'}[\mathrm{m}^2\ \mathrm{g}_{-\mathrm{catCFF}}{}^{-1}]}{W[\mathrm{wt\%}]/100} \qquad (1)$$

Here, W is the weight ratio of the loaded catalyst to the cat-CFF, and the specific surface area of α-Al_2O_3 is assumed to be negligibly small.

Methods A and C with the same calcination temperature but the different dipping condition gave comparable Sa″ values of 9.5 $m^2\ g^{-1}$ and 10.0 $m^2\ g^{-1}$, respectively, and the Sa″ value decreased monotonically with the increase in the calcination temperature. These results clearly show that the Sa″ value depends on the calcination temperature. Furthermore, when the calcinations temperatures were the same, Sa″ values of the cat-CFFs were close to the surface areas of LKM82 powder which was obtained by the evaporating-to-dryness process.

Fig. 7 shows TPO profiles of PM combustion with a bare CFF and selected cat-CFFs. CO_2 was formed as the main product in the temperatures range 300–800 °C (Fig. 7(A)). The formation of CO was also observed in the same temperature range of the CO_2 formation, but its amount was much lower than that of CO_2 formed, as shown in Fig. 7(B). Therefore, we can discuss the PM combustion performance of cat-CFFs from the CO_2 formation curves. In the case of PM combustion with the bare CFF, the amount of CO_2 increased with increasing the heat temperature, and the profile for the CO_2 concentration showed a maximum (T_C) at around 580 °C, which was close to the temperature of the self-combustion of CB without the assistance of a catalyst.

The coating of LKM82 catalyst on the CFF was effective to promote the PM combustion. When the cat-CFFs were used for the reaction, T_C shifted to lower temperature side and two peaks were observed for the CO_2 profiles (Fig. 7(A)). The lower-temperature peaks were ascribed to the catalytic combustion of PM, and the high-temperature ones were ascribed to non-catalytic combustion of PM. The T_C values of the lower-temperature peaks were different by *ca.* 100 °C, depending on the calcination temperature or the specific surface area of coated LKM82: the T_C values were 420 °C and 525 °C for Methods B and D, respectively. On the contrary, the T_C values of the high-temperature peaks were almost independent



(a) CFF without catalyst. (α-Al_2O_3)

(b) 3.52 wt%LKM82/CFF prepared by Method A[a].

(c) 3.17 wt%LKM82/CFF prepared by Method B[a].

(d) 2.58 wt%LKM82/CFF prepared by Method C[a].

(e) 2.98 wt%LKM82/CFF prepared by Method D[a].

Fig. 6. SEM images of surfaces of (a) bare ceramic foam filter and $La_{0.8}K_{0.2}MnO_3$-coated ceramic foam filters prepared by (b) Method A, (c) Method B, (d) Method C and (e) Method D. [a]See Table 3 for the details of the preparation method.

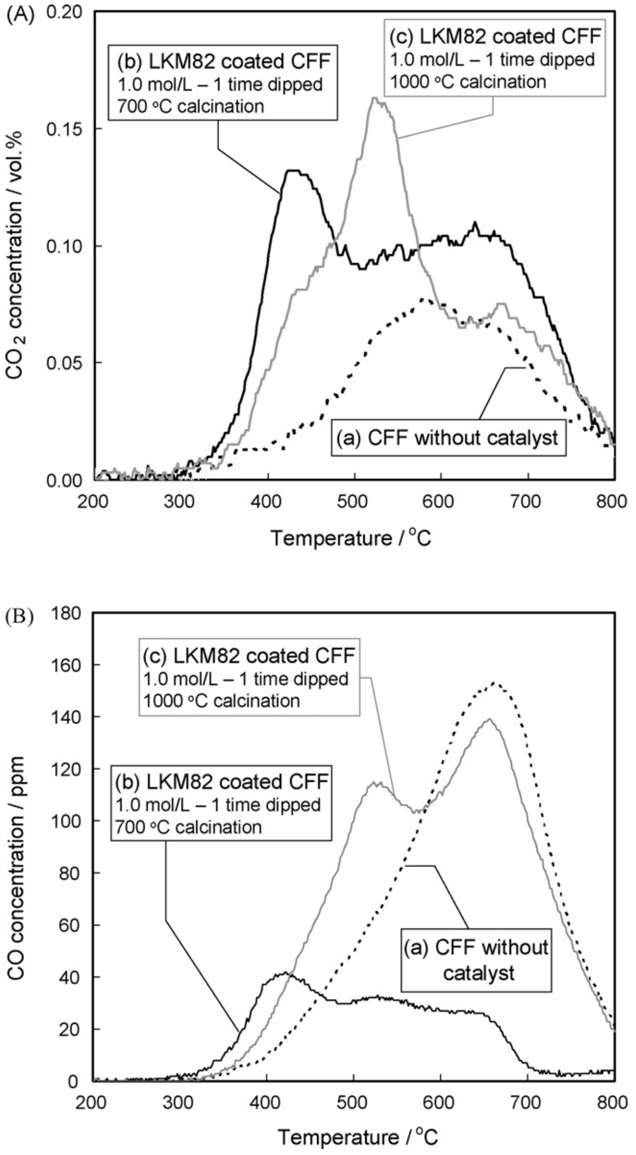


Fig. 7. Temperature programmed oxidation of PM trapped on ceramic foam filters with and without catalyst coating: profiles of (A) CO_2 and (B) CO concentrations. (a) Bare filter, (b) $La_{0.8}K_{0.2}MnO_3$-coated filter prepared by Method B, (c) $La_{0.8}K_{0.2}MnO_3$-coated filter prepared by Method D. See Table 3 for the details of the preparation method.

of the preparation conditions. These behaviors can be explained as follows. The catalytic combustion of the trapped PM takes place at the contact point of the PM and catalyst. The trapped PM particles which are directly contacted with or present close to the catalyst combust catalytically under the lower-temperature peak, and those remote from the catalyst combust non-catalytically under the high-temperature peak. These findings suggest that the catalytic effect for the PM combustion is confirmed with cat-CFFs and that the over-accumulation of PM to cause the non-catalytic combustion should be avoided for the catalytic removal of PM in high efficiency.

Fig. 8 shows the relationship between the surface area of the cat-CFFs (Sa′) and the PM combustion temperature; T_C values of the lower-temperature peaks were used for cat-CFFs. The T_C value decreased with the increase in Sa′, indicating that PM combustion performance of the cat-CFF was improved by increasing surface area of the cat-CFF. This finding is consistent with the observation obtained from the TG measurement which used powder catalysts of LKM82 and model substrate (CB), and further suggests that catalyst surface area controls the PM combustion performance even under the practical contact conditions between PM and catalyst.

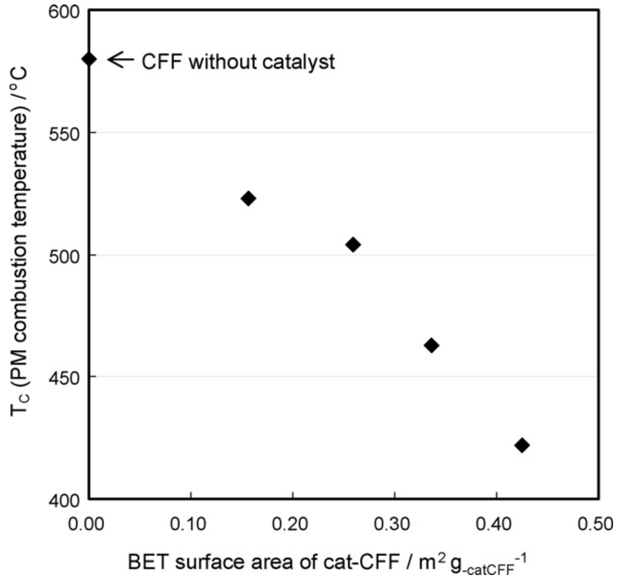


Fig. 8. The correlation between the PM combustion temperature (T_C) and specific surface area of the catalyst-coated ceramic foam filters.

Several researchers have reported the factors affecting the catalytic performance for soot oxidation including the redox properties of the catalyst, oxidants in gas phase (O_2, NOx, CO), and catalyst surface area [17,18]. Wang et al. investigated the catalytic properties of $La_{1-x}K_xMnO_3$ for diesel soot combustion under the loose-contact condition and suggested that these three factors affect the catalytic performance. They reported that the change of the $La_{1-x}K_xMnO_3$ composition lead to the decrease in soot combustion temperature by 70–80 °C under loose-contact condition [8]. It has been reported that the PM combustion temperature decreases with increasing the catalyst-to-PM ratio for Cu–K–V catalysts [19,20], and similar behavior was also observed for $La_{1-x}K_xMnO_3$ catalyst in our study (the data are not shown). These findings indicate that the increase in the number of contact points between the catalyst and PM promoted the PM combustion reaction. The present study was focused on the effect of surface area and revealed that the PM combustion performance strongly depended on the surface area even when both the overall composition of the catalyst and the catalyst-to-PM ratio were the same. It is likely that the number of contact points between the catalyst and PM increased by the increase in the surface area, which is the cause of improved catalytic performance for PM combustion.

4. Conclusion

In this study, $La_{0.8}K_{0.2}MnO_3$ catalysts with different surface areas were prepared and used for the PM combustion reaction under artificial tight- and loose-contact conditions and practical contact conditions. PM combustion temperature decreased *ca.* 150 °C and *ca.* 200 °C with increasing surface area from 1.1 $m^2\,g^{-1}$ to 18.5 $m^2\,g^{-1}$ under tight-contact and loose-contact conditions, respectively, although the composition of the catalysts were same. PM combustion performance of $La_{0.8}K_{0.2}MnO_3$ under the practical contact condition was also evaluated by using $La_{0.8}K_{0.2}MnO_3$-coated ceramic foam filters and the practical diesel exhaust gas, and the PM combustion temperature of the $La_{0.8}K_{0.2}MnO_3$-coated ceramic foam filter decreased by more than 100 °C with increasing surface area of the catalyst. These results strongly suggest that the surface area is one of the important factors controlling catalytic performance for PM combustion, and the improvement of contact points between the catalyst and PM is indispensable for the preparation of high performance catalysts.

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