Effect of Different Aging Conditions on the Soot Oxidation by Thermogravimetric Analysis

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ABSTRACT: Diesel particulate filter is an effective device to reduce diesel particulate emission. The particles in diesel particulate filter are usually affected by the aging of high-temperature exhaust gas before the regeneration process. In order to investigate the effect of aging conditions on the soot oxidation process, the effect of aging temperature and aging time on the oxidation process of carbon black (Printex-U, PU) and the PU/catalyst/ash mixture are studied by thermogravimetric analysis. The aging PU particles have lower starting temperature, peaking temperature, ending temperature, and activation energy. Compared with the particles without aging, the PU particles with a 400 °C aging temperature and 20 h aging time are able to reduce the activation energy from 191.2 to 158 kJ/mol. Low aging temperatures (200–300 °C) and the catalyst have a certain synergistic effect on the improvement of PU oxidation activity. The PU/CeO2 mixture with a 300 °C aging temperature and 20 h aging time decreases the activation energy from 178.4 to the lowest 113.6 kJ/mol. The addition of CaSO4 in PU particles cannot stop the improvement of its oxidation activity by aging, but it reduces the effect of aging. This work is helpful to reveal the mechanism of aging on PU and the PU/catalyst/ash mixture in air environment.

1. INTRODUCTION

A diesel engine is favored by people because of the high thermal efficiency, good fuel economy, strong power, and high reliability.1,2 However, diesel engines emit more particulate matter (PM) than gasoline engines, which is harmful for human health and atmospheric environment, so it is needed to take some steps to deal with it.3,4 Nowadays, diesel particulate filter (DPF) has become an indispensable part of a diesel after treatment system, and it is an important method to capture PM with more than 95% capture efficiency.5,6

The pressure drop in DPF increases with PM depositing in DPF, which affects the performance of a diesel engine, so DPF needs to be regenerated.7,8 There are two main types of DPF regeneration, which includes active regeneration and passive regeneration. The electrical heater or post-fuel injection strategy is used to increase the DPF inlet temperature to 550 °C or higher for the active regeneration. Passive regeneration is able to reduce the regeneration temperature to 200–400 °C with the help of catalysts.9,10 Passive regeneration is the coating of a layer of catalyst in DPF to reduce the starting temperature of PM, so it can be regenerated at the exhaust temperature of the engine.11,12 In the process of active regeneration, PM is not regenerated immediately after accumulating in DPF, which is subjected to the aging effect of exhaust gas for a certain period of time, and the exhaust temperature of the engine is usually between 180–400 °C.13

When the exhaust back pressure of DPF reaches a certain degree, the regeneration process will start.14 Aging not only affects the performance of DPF but also affects the properties of PM and the catalyst deposited in DPF. PM usually includes carbon, ash, organic compounds, and sulfate materials.15–17 It is found that CeO2 is a good catalyst for soot, which is able to reduce the starting temperature of soot greatly.18–20 Ash usually consists mainly of Ca, Mg, P, Zn, S, O, and minor Fe, Al, Si, and CaSO4 can be found in soot as a kind of ash.21 Some experimental and numerical studies have been conducted to understand the effect of aging on DPF and PM. Zhang et al.22 applies that the maximum wall temperature and the pressure drop are taken as the evaluation indexes of the effect of thermal aging on DPF and compared the effects of four structural factors (wall thickness, mean pore size, porosity, and channel width) and five operational factors (exhaust flow rate, exhaust oxygen concentration, microwave power, catalytic additive mass concentration, and the mass of ash deposition) on the thermal aging of DPF. DaCosta et al.23 concludes that aging at 550 or 650 °C for 100 h has no significant effect on the filtration efficiency, pressure drop, and regeneration efficiency of DPF. Dimitrios et al.24 reveals that ash aging improves filtration efficiency and reduces filtration
pressure drop of DPF. Dimitrios et al.25 indicates that the performance of catalytic diesel particulate filter (CDPF) with cerium-based oxide as the catalyst decreased significantly after aging at 800 °C and 21% O2 for 10 h. Cheng and Lehmann26 reports that the aging of carbon black will increase the surface oxygen concentration and decrease the carbon concentration in the range of −22 to 70 °C. Meng et al.27 points out that the hollow and disordered microcrystalline structure in soot particles can be removed by heat or oxidation treatment, which is helpful to reduce the activation of soot particles. The above studies explore that the thermal aging has little effect on the performance of DPF but has a great effect on the soot particles in DPF. However, the above studies are not enough to explain the effect of thermal aging on soot and the soot/ash/catalyst mixture.

The novelty of this study is to explore the effect of different aging conditions on the oxidation performance of soot according to the real condition of the diesel exhaust temperature and the DPF regeneration cycle and discuss the effect of aging and catalyst/ash coexistence. This work lays a foundation for the study of the influence mechanism of aging on soot and provides a certain direction for the improvement of the DPF regeneration strategy in the future.

2. DESCRIPTION OF EXPERIMENTS

2.1. Experiment Materials. PU is widely used to replace diesel engine emission particles because of its similar physical and chemical properties to diesel engine emission particles.28−31 It is well known that thermogravimetric analysis is a method32 to reflect the oxidation properties of substances, because the degree of weight loss during heat treatment can be represented by the TGA diagram.33−35 The major properties of PU (Degussa GmbH, Frankfurt, Germany) employed in TGA tests are shown in Table 1. A fixed bed reactor is used to provide different aging conditions for PU and PU/catalyst/ash mixtures, and its main parameters are shown in Table 2. The influence of different aging conditions on the soot oxidation process have been performed by TGA (TG209F3 from NETZSCH, Germany), and the main parameters of TGA are listed in Table 3. CeO2 is selected as the catalyst and CaSO4 is selected as the model ash in this experiment. The characteristic parameters of CeO2 and CaSO4 are shown in Table 4.

Table 1. Physical Properties of Carbon Black

| soot | diameter (nm) | BET (m²/g) | oil absorption (g/100 g) | ash content (%) |
|------|---------------|------------|--------------------------|-----------------|
| Printex U | 25 | 92 | 460 | 0.02 |

Table 2. Main Parameters of the Fixed Bed Reactor

| project | specification |
|---------|---------------|
| equipment type | TF1200−60 |
| rated power | 3 kW |
| rated voltage | 220 V |
| equipment weight | 45 kg |
| temperature range | ≤1100 °C |
| factory number | NB800009 |
| outline size | 960 × 600 × 500 mm |
| heating element | resistance wire |

Table 3. Main Parameters of TGA

| parameters | value |
|------------|-------|
| measuring dynamic range (g) | 0−2 |
| balance sensitivity (μg) | 0.1 |
| heating rate (°C/min) | 0.001−100 |
| size of crucible (mm) | Φ6.8 |
| volume of crucible (μL) | 268 |
| range of temperature (°C) | 45−1000 |

Table 4. Physical Properties of CeO2 and CaSO4

| components | diameter (nm) | metal basis (%) |
|------------|--------------|----------------|
| CeO2 | 20−50 | 99.5% |
| CaSO4 | 50 | 99.9% |

2.2. Experimental Method. The PU and PU/CeO2/CaSO4 mixture with or without the aging process are used in the TGA experiment. The mass of PU selected in each test is 3 mg. The summary of characteristic parameters for all the cases of this study is shown in Table 5. According to the exhaust temperature and active regeneration cycle, the aging temperature range of this experiment is 200 °C−400 °C, and the aging time is 5−30 h. The addition of a catalyst has a good promoting effect on the combustion of soot. The weight ratio that is selected is 1:5 for PU/CeO2.36 Only a small part of ash usually exists in soot, so the mass ratio of PU/CaSO4 is 1:1.37

First, 1 g of PU or PU/CeO2 and PU/ CaSO4 mixtures are put into the center of the glass tube of the fixed bed, and the fixed bed system diagram is shown in Figure 1. Then, open both sides of the glass tube, and let the air flow naturally. The whole experiment is aged under the condition of air circulation. The fixed bed can be raised to different temperatures and kept for a certain period of time through adjustment.

The schematic diagram of TGA is shown in Figure 2, and the weight of PU is fixed at 3 mg in each test. PU or mixture particles are put into the oxidation crucible, and the temperature rises from 45 to 800 °C and maintains at 800 °C for 10 min. The inner diameter and the height of the alumina crucible are 6.8 and 7.4 mm, respectively. The heating ramp, total flow rate, and oxygen concentration are 10 °C min−1, 100 mL min−1, and 10%, respectively. Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves are obtained by experiments. TG curves are obtained by continually recording the mass loss with the increasing temperature, and DTG curves are achieved by differentiating the TG curves. The accuracies of the TGA for the mass and temperature are 0.1 μg and 0.1 °C, respectively (Figure 3).

2.3. Data Analysis. The activation energy for the oxidation of soot is calculated by the differential method. First, the conversion rate (α) of the sample is defined as38,39

\[ \alpha = \frac{m_0 - m}{m_0 - m_\infty} \tag{1} \]

where \( m_0 \) is the mass of sample at the beginning of the reaction, \( m \) is the mass at the end of the reaction, and \( m_\infty \) is the sample mass at \( T(I) \), the reaction temperature, and time, respectively.

The relative reaction rate γ is expressed as40

\[ \gamma = \frac{dx}{dt} = A' \cdot \exp\left(-\frac{E}{RT}\right) \cdot (1 - \alpha) \cdot S \cdot \text{PO}_{2} \tag{2} \]
where \(A', E, n, S_g\) and \(p_{o2}\) represent the pre-exponential factor, activation energy, reaction order, specific surface area of soot, and oxygen partial pressure, respectively.

The heating rate \(\beta\) of TGA is defined as:

\[
\beta = \frac{dT}{dt}
\]

Substituting eq 3 into eq 2, separating variables, and then logarithm to both sides:

\[
\ln \frac{d\alpha}{dT(1 - \alpha)^n} = \ln \left( \frac{A'S_g p_{o2}}{B} \right) - \frac{E}{RT}
\]

The \(\frac{d\alpha}{dT(1 - \alpha)^n}\) and \(1/T\) are linearly related, and \(E\) can be obtained by obtaining its slope.

### Table 5. Summary of Characteristic Parameters in all of the Cases of this Study

| case | soot/catalyst/ash wt ratio | aging temperature (°C) | aging time (h) | \(T_h\) (°C) | \(T_p\) (°C) | \(T_s\) (°C) | \(E\) (kJ/mol) |
|------|---------------------------|------------------------|---------------|--------------|--------------|--------------|----------------|
| 1–2  | PU/—/—                    | 400                    | 5             | 580 ± 4      | 643 ± 1      | 678 ± 2      | 191.2 ± 2.5   |
| 3–4  | PU/—/—                    | 400                    | 10            | 555 ± 2      | 643 ± 1      | 678 ± 2      | 176.5 ± 5.9   |
| 5–6  | PU/—/—                    | 400                    | 20            | 550 ± 3      | 642 ± 0      | 668 ± 4      | 160.5 ± 4.1   |
| 9–10 | PU/—/—                    | 400                    | 30            | 539 ± 3      | 628 ± 3      | 658 ± 2      | 158.4 ± 2.6   |
| 11–12| PU/—/—                    | 200                    | 20            | 573 ± 5      | 643 ± 1      | 680 ± 3      | 180.3 ± 5.7   |
| 13–14| PU/—/—                    | 300                    | 20            | 561 ± 5      | 643 ± 1      | 677 ± 4      | 176.4 ± 1.2   |
| 15–16| PU/—/—                    | 350                    | 20            | 553 ± 5      | 642 ± 1      | 665 ± 3      | 160.1 ± 4.7   |
| 17–18| PU/—/—                    | 1:5                    | 603           | 510 ± 5      | 606 ± 1      | 608 ± 1      | 178.4 ± 0.3   |
| 19–20| PU/CeO\(_2\)/—           | 1:5                    | 200           | 500 ± 4      | 653 ± 2      | 610 ± 6      | 167.4 ± 5.9   |
| 21–22| PU/CeO\(_2\)/—           | 1:5                    | 250           | 485 ± 3      | 572 ± 3      | 607 ± 5      | 129.9 ± 5.5   |
| 23–24| PU/CeO\(_2\)/CaSO\(_4\)  | 1:5                    | 300           | 476 ± 4      | 555 ± 3      | 604 ± 4      | 113.6 ± 9.1   |
| 25–26| PU/CeO\(_2\)/CaSO\(_4\)  | 1:1                    | 300           | 578 ± 7      | 644 ± 4      | 684 ± 8      | 190.4 ± 0.4   |
| 27–28| PU/—/—/CaSO\(_4\)        | 1:1                    | 350           | 574 ± 7      | 642 ± 4      | 680 ± 8      | 187.1 ± 2.1   |
| 29–30| PU/—/—/CaSO\(_4\)        | 1:1                    | 350           | 566 ± 6      | 638 ± 3      | 676 ± 7      | 182.2 ± 1.2   |
| 31–32| PU/—/—/CaSO\(_4\)        | 1:1                    | 400           | 555 ± 5      | 627 ± 2      | 656 ± 5      | 177.8 ± 7.6   |
| 33–34| PU/—/—/CaSO\(_4\)        | 1:1                    | 400           | 555 ± 5      | 627 ± 2      | 656 ± 5      | 177.8 ± 7.6   |

The starting temperature \(T_s\), peaking temperature \(T_p\), and ending temperature \(T_h\) are determined by Figure 1. The lower the \(T_s\) is, the easier the soot is to ignite, which is beneficial to the active regeneration of DPF.

### 3. RESULTS AND DISCUSSION

#### 3.1. Effect of Aging Time on Oxidative Combustion

The effect of four different aging times on the oxidation performance of PU are compared at the 400 °C aging temperature, and the TGA curves are shown in Figure 4. The reason why the 400 °C aging temperature is chosen is that the higher the temperature is, the more the PU particle structure will be affected. From TGA curves, the mass loss of PU can be roughly divided into two stages. The first stage is at 0–200 °C, and the particles lose weight slowly and the curve goes down by 5% at this stage, which mainly occurs in the evaporation of water in PU. The second stage is at 200–800 °C, which causes the oxidation reaction of PU particles to occur, and the TGA curves to decrease rapidly. The TGA curves of PU particles after aging tend to shift toward the low-temperature zone. The longer the aging time is, the more obvious the curve deviates, which indicates that the aging PU particles are easier to burn. The curve tends to be stable when the aging time is greater than 20 h.
Figure 5 compares the starting and peaking temperatures of PU particles in different aging times at the 400 °C aging temperature. Aging is beneficial to reduce the starting and peaking temperatures of PU particles. The starting temperature of PU particles decreases significantly at the 5 h aging time, which drops from 580 to 555 °C. With the increase of aging times, the two kinds of temperatures have different degrees of decline. When the aging time is 20 h, the starting temperature drops to the lowest 538 °C.

The activation energies of PU particles in different aging times at the 400 °C aging temperature are shown in Figure 6. The curve shows a trend of decreasing first and then stabilizing. The activation energy decreases significantly from 191.2 to 160.5 kJ/mol within 10 h of aging time. When the aging time is more than 10 h, the change of activation energy is not significant. The lowest activation energy is 158 kJ/mol at the 20 h aging time. It indicates that the energy required for the oxidation reaction of PU particles after aging is reduced, which is beneficial to the DPF regeneration process.

In general, the extension of aging time is beneficial to the active regeneration of DPF. The starting temperature and activation energy of PU particles are reduced, which means that the DPF regeneration process can be completed only at a lower temperature and in a shorter time. Aging may change the internal structure of PU particles, which increases activity of their reaction with oxygen. Fang et al. report that thermal aging is able to change the structure of PU and increases its disorder degree and reduces its graphitization structure. PU with a low graphitization structure has higher oxidation activity and is easier to react with oxygen. The aging PU particles burn more easier once they are heated again.

Figure 7 shows the comparison of the starting and peaking temperature of PU with different aging temperatures under the 20 h aging time. Aging temperature has a greater impact on the $T_s$, but it has a small impact on the $T_p$ when the aging temperature is greater than 350 °C. It is effective for the DPF regeneration process to increase the aging temperature, which provides a reference for future improvement of the DPF regeneration strategy.

Furthermore, the activation energy of different aging temperatures is calculated, and the results are shown in Figure 9. The change of activation energy can be roughly divided into two stages. The first stage is below 300 °C, and there is no obvious difference between the aging curve and the non-aging curve. Both curves shift to the low temperature zone to a large extent when the aging temperature is more than 300 °C. The higher aging temperature is the more obvious the shift is. It indicates that a high aging temperature (300−400 °C) is more likely to affect the oxidative activity of PU particles.

Figure 8 shows the comparison of the starting and peaking temperature of PU with different aging temperatures under the 20 h aging time. Aging temperature has a greater impact on the $T_s$ but it has a small impact on the $T_p$ when the aging temperature is greater than 350 °C. It is effective for the DPF regeneration process to increase the aging temperature, which provides a reference for future improvement of the DPF regeneration strategy.
two stages, which is similar to the TGA curves. The thermal aging process has little effect on the activation energy at 200 °C. The higher the aging temperature is, the more obvious the activation energy decline is.

In general, the higher the aging temperature is, the more beneficial to increase the oxidation activity and the combustion of PU particles. The higher the temperature is, the more the structure of PU particles changes, which will change the structure of PU particles from crystalline carbon to amorphous carbon. This means that the structure of carbon black tends to disorder more. The disordered PU particles are easier to react with oxygen, which is consistent with the results of this experiment.

3.3. Effect of Aging on Oxidative Combustion of PU under the Catalyst Condition. In this section, the thermal aging process is investigated when catalysts are added. The catalyst is CeO$_2$, and the weight ratio of PU to CeO$_2$ is 1:5. The aging time is 20 h, and the aging temperatures are 200, 250, and 300 °C, respectively.

The TGA curves obtained from the experiment are shown in Figure 10. CeO$_2$ has a good catalytic effect, and the TGA curves after the addition of CeO$_2$ are significantly shifted to the low temperature zone. Furthermore, the curve is further shifted to the low temperature zone when the catalyst and aging act, and this indicates that aging under 300 °C will not destroy the structure of the catalyst but jointly promote the oxidation activity of PU particles.

Figure 11 compares the starting and peaking temperatures of the PU/CeO$_2$ mixture at different aging temperatures. From Figure 11, two kinds of temperatures decrease obviously, $T_s$ decreases from 580 to 510 °C, and $T_p$ decreases from 643 to 569 °C after adding CeO$_2$ into PU. Both temperatures are further reduced when the mixture is aged, and $T_s$ decreases to the lowest 476 °C at the aging temperature of 300 °C. It is beneficial for DPF regeneration control, because active regeneration usually involves increasing the DPF inlet temperature through fuel injection, so reducing the ignition temperature can decrease the fuel consumption.

The activation energy of the mixture at different aging temperatures is further calculated, as shown in Figure 12. The overall trend of the activation energy curve is gradually decreasing. Moreover, the activation energy drops to the lowest 113.6 kJ/mol at the 300 °C aging temperature, which reduces by nearly half. The above phenomenon indicates that thermal aging and the catalyst can jointly promote the combustion of PU particles.

In general, both thermal aging and CeO$_2$ can be used for DPF regeneration control. The CeO$_2$-based catalysts have remarkable activity for soot oxidation due to their ability to switch between Ce$^{3+}$ and Ce$^{4+}$ states by the high oxygen storage capacity performance. It is reported that the structure of CeO$_2$ begins to be destroyed above 1000 °C, but the catalytic effect did not change obviously at a low temperature, so the catalytic effect and low temperature...
thermal aging can jointly promote the combustion of PU particles.

3.4. Effect of Aging on Oxidative Combustion of PU under the Ash Condition. Particles deposited in DPF usually contain a small amount of ash. In this section, it is further explored whether aging has the same effect on PU/CaSO$_4$ mixtures. The ash is CaSO$_4$, and the weight ratio of PU to CaSO$_4$ is 1:1. (the aging conditions: time is 20 h, the aging temperatures are 300, 350, and 400 °C) The aging temperature starting from 300 °C is adopted, because low temperature aging has no obvious effect on the PU/CaSO$_4$ mixture.

The TG and DTG curves obtained from the experiment are shown in Figure 13. The presence of a small amount of CaSO$_4$ will not significantly affect the combustion of PU particles, and its curve roughly coincides with the original curve, which is similar to previous results. $^{53,54}$ With the increasing aging temperatures, the TGA curve of the mixture shifts to the low temperature zone, which indicates that the presence of ash cannot hinder the improvement of thermal aging on PU oxidation activity.

The experimental $T_s$ and $T_p$ are shown in Figure 14. After adding CaSO$_4$ into PU, the $T_s/T_p$ shows little changes and stabilizes at 580 °C/644 °C, respectively. The aging has the same effect on the decrease of the $T_s/T_p$ of the mixture as in the second section, and the $T_s/T_p$ decreases to the lowest 555 °C/627 °C at 400 °C. The results show that the ash does not affect the effect of aging on the reduction of the starting temperature of particles, which provides a reference for the improvement of the DPF regeneration strategy.

Figure 15 shows that the comparison of the activation energy of the PU/CaSO$_4$ mixture at different aging temperatures. The curve generally shows a slow downward trend. After the addition of CaSO$_4$ in PU, the activation energy does not change much, which is similar to the previous results. However, its activation energy decreases from 191.2 to 177.8 kJ/mol with the effect of aging. Aging also affects the internal structure of the mixture, which is leading to a decrease of the activation energy.

In general, when PU contains a small amount of CaSO$_4$, aging continues to affect the structure of PU and improve its oxidative activity. However, the aging effect of the mixture is not as good as that of pure PU, and the activation energy/$T_s$ only drops to 177.8 kJ/mol/555 °C. It is due to ash whose particle diameter is large has not catalytic effect, and mixing with PU may cause PU cannot be uniformly heated, and the effect of thermal aging will decrease.

4. CONCLUSIONS

The oxidation characteristics of PU particles under different aging conditions are studied by means of fixed bed and TGA. The following results are obtained based on above experiments and analysis:

**Figure 12.** Comparison of $E$ of the PU/CeO$_2$ mixture at different aging temperatures.

**Figure 13.** Comparison of TGA curves of the PU/CaSO$_4$ mixture at different aging temperatures.

**Figure 14.** Comparison of $T_s$ and $T_p$ of the PU/CaSO$_4$ mixture at different aging temperatures.

**Figure 15.** Comparison of $E$ of the PU/CaSO$_4$ mixture at different aging temperatures.
The aging PU particles have higher oxidation activity and are easier to ignite compared to the non-aging PU particles.

With the increase of aging times from 0 to 20 h at 400 °C, the oxidation activity of PU particles increases first and then stabilizes, and the activation energy decreases from 191.2 to 158.4 kJ/mol.

The higher the aging temperature is, the higher the oxidation activity of PU particles is.

When the aging temperature is in the range of 200–300 °C, adding CeO₂ to PU and thermal aging of PU have a certain synergistic effect, which can jointly improve the oxidation activity of PU. With the increase of the aging temperature from 200 to 300 °C at a 20 h aging time, the activation energy of the PU/CeO₂ mixture decreases from 178.4 to 113.6 kJ/mol.

When a small amount of CaSO₄ exists in PU, it does not stop the increase of oxidation activity of PU by aging.

The practical significance of this work is to explore the effect of aging on soot and a soot/catalyst/ash mixture in an O₂/N₂ atmosphere, which can be used in a regeneration process. In the future, the effect of aging on soot under more aging conditions and real engine exhaust conditions will be explored.

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**Notes**

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