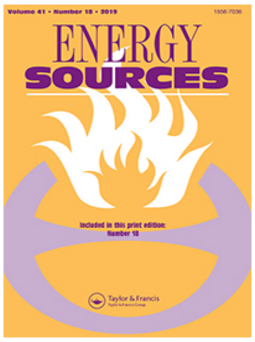

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Ash deposited in diesel particular filter: a review

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ABSTRACT
Diesel particle filter (DPF) is widely considered as the most effective approach to reduce and control particle matter (PM) emissions. However, with the continuous accumulation of particles, DPF has poor performance and has to burn PM, i.e. regeneration. Unlike soot, ash cannot be removed from DPF through burning, which shows a negative effect on DPF performance. Therefore, the recent research progress about ash deposited in DPF has been summarized. The chemical composition and formation process were analyzed. The effects of ash on the DPF performance were addressed. The results show that the ash is mainly generated from lubricating oil, and the chemical composition of ash mainly consists of Ca, Mg, P, and S. Meanwhile, the ash morphology and color depended on the ash chemical composition. The flow-induced transport and the regeneration-reduced transport alter the mobility of the ash from the channel walls to the plug. The ash cannot influence the DPF performace in the nomal condition while it has an important effect on back pressure and soot oxidation characteristics. Furthermore, there are still disagreements on the mechanism of the ash distribution process and the effect of the distribution form of ash deposits in the DPF channel on the pressure drop. In the future, the investigations on the ash physico-chemical property and the ash formation process will be helpful to elucidate the abovementioned problems. In addition, in order to reduce the ash deposited and ensure the DPF normal operation, the more strict lubricant regulations should be implemented in China.

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Ash; particle matter filter; regeneration; soot; emission control

Introduction

As one of the most important power equipment, diesel engine has attracted considerable attention, owing to its lower fuel consumption and lower carbon dioxide emissions. Nevertheless, particle matter (PM), which is a main concern in development of diesel engine, has a negative effect on human health problems and environment pollutions (Chen, Ibrahim, and Wang 2014; Fang et al. 2017; Kagawa 2002; Kittelson 1998; Wang et al. 2016; Yang, Wang, and Wu 2017b; Zhang et al. 2017). Hence, several approaches consisting of diesel particle filter (DPF) (Yu, Luss, and Balakotaiah 2013), improvement of fuel quality (Liu et al. 2008; Wu et al. 2017), and internal purification technology (Knecht 2008) have been put forward to reduce PM emissions to match the more stringent environmental regulations. Among them, DPF is widely considered as the most effective

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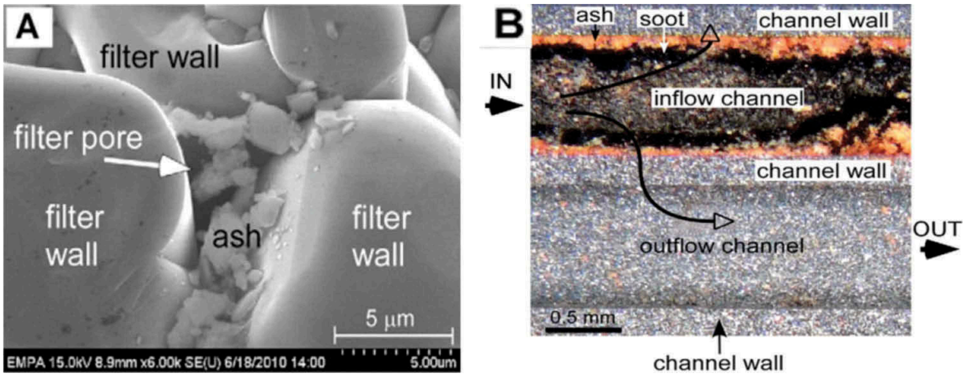

Figure 1. (A) SEM image of ash agglomerates in the pores of the channel wall of DFP. (B) Schematic diagram of ash layer (reddish) and soot layer (black) (Liati and Dimopoulos Eggenschwiler 2010; Liati et al. 2012a).

approach to reduce and control PM emissions because the PM removal efficiency is more than 98% (Gao et al. 2018).

Hitherto, numerous investigations have focused on the DPF, including DPF regeneration (Bensaid and Russo 2011; Fang et al. 2017; Stepien et al. 2015; Yang et al. 2017a; Zhang et al. 2016b), DPF integration with other after treatment components (Guan et al. 2015; Naseri et al. 2011), the effect of DPF on diesel engine (Bikas and Zervas 2007; Yu et al. 2017), and mathematics simulation of DPF (Koltsakis and Stamatelos 1997; Yamamoto and Yamauchi 2013). When it comes to the DPF regeneration, PM accumulation will increase filter back pressure, decrease fuel economy, and weaken engine performance. Therefore, DPF has to be regenerated by burning or oxidizing trapped PM. Unlike to soot particles, ash cannot be removed from DPF by burning and will deposit in it, as shown in Figure 1. In consideration of the properties both of ash and DPF, the ash shows a negative effect on DPF performance, such as reducing soot storage capacity, increasing DPF pressure drop, decreasing fuel economy, and shifting the balance point temperature during regeneration (Kamp et al. 2014). Therefore, the recent research progress about ash deposited in DPF has been summarized.

In this work, a comprehensive overview of ash deposited in the DPF was reviewed. To begin with, the chemical composition and the formation process of ash were concluded. Furthermore, the influences of ash on DFP performance were analyzed. Finally, the limitation and the future trend of ash research were also pointed out.

Source and chemical composition

It is generally recognized that ash is mainly produced from metals in the diesel fuel, engine wear and corrosion, lubricating oil additives, and lubricating oil (Sappok and Wong 2007; Tornehed and Olofsson 2011). Among them, lubricating oil is considered the main source and accounts for 90% (mass fraction) (Bagi, Bowker, and Andrew 2016). Bardasz et al. (2005) found that the amount of ash collected by the DPF was directly related to the oil consumption. Furthermore, Aravelli et al. (2007) indicated that the amount of ash deposited in the DPF was related to the accumulation of lube oil consumption and the correlation coefficient is 0.9975. Based on the literature review, it should be noted that only a little fraction of lube oil ash deposited into the DPF which was about 20%–70% (Tornehed and Olofsson 2011). The others may deposit in piston, chamber-wall surface, exhaust pipe wall, oil filter, oil pan, and the lose because of evaporating. In addition, it is worth noting that the ash accumulation deposited in DPF is quite little in the real-world operations and a long time should be taken to get a large amount of ash. In detail, more than 80% of material deposited in DPF is ash after 241,000 km (150,000 miles) and several thousand hours are required to observe significant ash (Sappok and Wong, 2010b). Consequently, in order to investigate conveniently and address this gap, two main methods are used, i.e. burning mixed lubricating oil and adding external acceleration of the consumption of lubricating oil system.

Investigation on the ash morphology and properties is necessary to understand the ash. Bagi et al. (2016) collected the samples from commercial DPF cleaner for servicing and employed inductively coupled plasma-optical emission spectrometry (ICP-OES) to achieve elemental concentration test. Liati et al. (2012a) carried out the chemical analyses of ash from a light truck DPF and a passenger car by using ICP-OES. Both results provided useful information about the elements distribution. As illustrated in Figure 2, the ash colors vary from the initial light yellow color and khaki to aurichalceous and to the final dark brown in appearance. With the exception of O element, the large proportion elements exited in some ash samples are Ca, P, S, Mg, Fe, and Zn, which often present in forms of $CaSO_4$, $Zn_3(PO_4)_2$, $FePO_4$, and $Ca_3(PO_4)_2$ and sulfides of Ga, Mg, and Zn, respectively, as shown in Figure 3. The similar results are also reported by the other works (Nemoto et al. 2004; Liati et al., 2012a; Gysel et al. 2014; Serrano et al. 2014). Moreover, Al, Cr, Ni, Cu, Mn, and cordierite composition and Pt/Pd derived from DPF are less in some ash samples. Based on the aforementioned presentations, Ca, Zn, S, and P mainly come from lubricant and mitigation of the metal

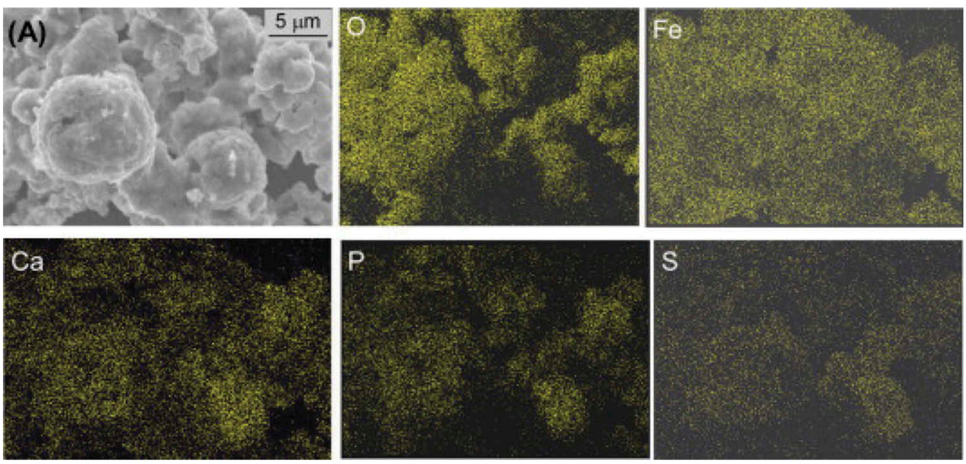

Figure 2. The morphology and elements distribution of ash (Liati et al. 2012a).

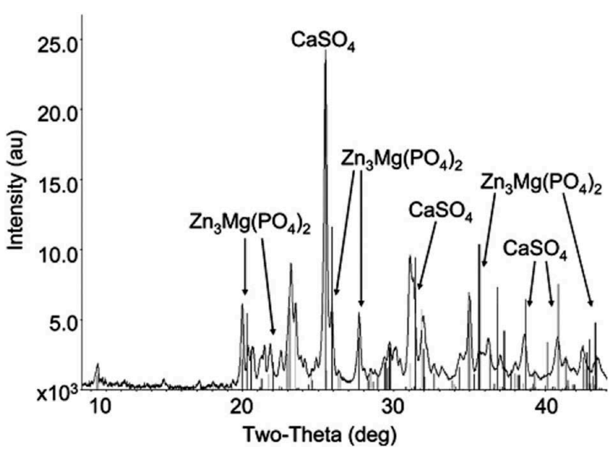

Figure 3. XRD spectra of ash deposited in DPF (Sappok and Wong 2010b).

content in lubricant and improvement of the lubricant quality can reduce the ash accumulation. The American Petroleum Institute, The European Automobile Manufacturers' Association, and Japanese Automotive Standard Organization proposed "Chemical Box" for the engine oil (Mc Geehan 2004), which requires the sulfur, sulfate ash, and phosphorus levels that are less than 0.3–0.5%, 1.0%, and 0.12%, respectively. However, relevant regulations about sulfur, sulfate ash, and phosphorus levels cannot be found in the GB11122-2006 in China. Thus, in order to reduce the ash deposited and ensure the DPF normal operation, the more strict lubricant regulations should be implemented to meet the DPF operational requirement in China VI emission regulation. In addition, ash elements have an impact on the morphology of ash (Kamp et al. 2014). As shown in Figure 2a, irregular agglomerates exist in ash sample which have round outlines and hollow shells. Ca-based lube oil ash shows smaller micro-scale while Mg-based lube oil ash shows bigger. Furthermore, the ash shows an extremely branched structure when zinc dialkyldithiophosphate additives are added into the lubricant.

The formation process of ash

Ash formation and distribution are characterized by a dynamic process that changes with the time and the amount of ash deposited in DPF. It is worth noting that the ash agglomeration and growth (as illustrated in Figure 4) occur quite rapidly, which only needs a single regeneration operation (Kamp et al. 2014). With the ash continuous accumulation, as shown in Figure 5, ash layer and plug ash are the main distribution forms deposited in DPF. After the formation of ash layer, a clear boundary between ash layer and channel is observed and particles should not penetrate into the ash layer. Thus, a distinct interface between ash layer and soot layer is also observed, as shown in Figure 1 (Kamp, Sappok, and Wong 2012). Hitherto, the publications focused on the mechanism of the ash distribution process are still controversial. On the one hand, Ishizawa et al. (2009) found that the mechanism depended on the DPF regenerative way. For the active regeneration, accumulation at the inlet channel of the plug was observed while the ash layer was formed with the passive regeneration. Meanwhile, micro size is bigger by using active regeneration which means permeability is better because a large amount of particle is burned in active regeneration and tends to be a cluster. As a result, the ash accumulates at the inlet channel of the plug with peeling off from DPF. In addition, higher regeneration temperature causes greater ash size, which is consistent with the other reports (Kamp, Sappok, and Wong 2012; Viswanathan, Rakovec, and Foster 2012). On the other hand, the attractive forces that existed in interparticles will influence the ash formation. Based on the results of in-situ optical system, Sappok et al. (2013) proposed that inspite of the

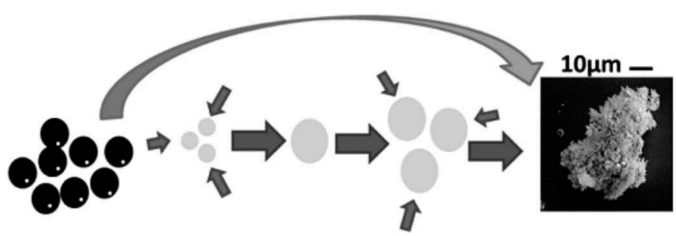

Figure 4. Schematic diagram of the agglomeration and growth (Kamp et al. 2014).

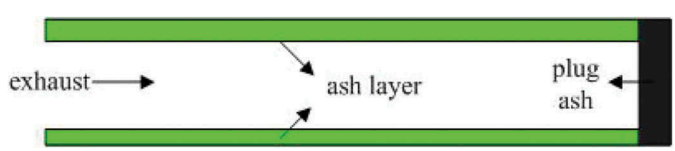

Figure 5. Schematic diagram of ash form existed in DPF.

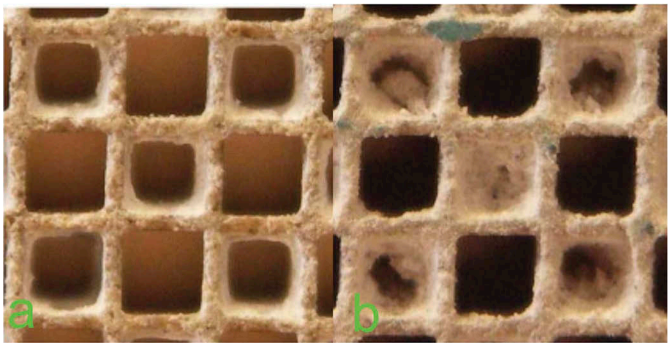

Figure 6. Ash (42 g/L) accumulation in DPF, (a) 57 mm from DPF face,(b) 133 mm from DPF face (Sappok and Wong 2010b).

absence of any flow through the channel, the process of oxidizing the soot was able to pull some of the ash away from the channel walls and eventually resulted in the formation of the plug ash at 600°C due to the presence of substantial cohesive. Moreover, Kamp et al. (2014) demonstrated that attractive forces for soot-soot, ash-ash, and soot-ash increased with soot or ash agglomerate size while attractive forces for soot-DPF and ash-DPF declined with soot or ash agglomerate size. Thus, particles could peel off from DPF and accumulate due to the inter-particle attraction and then result in the formation of large ash. Although the different mechanisms of the ash distribution process are demonstrated, the debate is still speculation which is attributed to the complex DPF regeneration, the intricate ash formation and accumulation, and the unclear fundamental mechanisms of ash property. Therefore, the investigations on the ash physico-chemical property and the ash formation process by using advanced instruments and theoretical derivation will be helpful to address the abovementioned problems. And it is assumed that if commercially available software such as VASP, Gaussian, DMol, and PWSCF based on the density functional theory are successfully combined with experimental process, the related conclusions will benefit to the understand the ash issues.

As shown in Figure 6, significant plug ash is observed near the back of the filter than the location near the front of the filter. Hitherto, the mechanisms altering the mobility of the ash from the channel walls to the plug are flow-induced transport and regeneration-reduced transport (Sappok et al. 2014). Based on the investigation for the microparticle detachment from the surface, there exists vertical lift force, fluid drag force, gravitational force (negligible), and adhesive force between microparticle and filter surface (Ibrahim, Dunn, and Brach 2004). Obviously, the rising fluid drag force or reducing adhesive force could make themicroparticles dislodge from the surface. With the airflow increasing and drag force increasing, the accumulated ash formed will migrate to the plug. In addition, Sappok et al. (2013) found that reducing the contact area between the soot and DPF surface resulted in the reducing of adhesive force between the soot and DPF surface during the regeneration process. Eventually, the accumulated soot was oxidized at the bottom and the plug ash formed.

The influences of ash on DFP performance

As we know, uncontrolled regeneration leading to excessive temperature will make the DPF substrate melt and the washcoat devitalization. Meanwhile, Choi et al. (2009) proved that in the presence of ash materials deposited in SiC-DPF, glazing, melting, and pinholes occurred under the air condition at 1100°C and the similar phenomenon also occurred at 800–900°C in the case of the presence of 10% H_2O in air or alkali metal deposited in SiC-DPF. For the cordierite, DPF performance would not be compromised by ash as long as the operating temperature did not exceed 900°C (Pomeroy et al. 2012). A similar result has been demonstrated (Merkel, Cutler, and Warren 2001). Generally, the diesel exhaust temperature is lower than 600°C in the nomal engine operating condition; thus, it is inferred that the ash cannot cause the DPF devitalization in the normal condition. The major challenge for the DPF survivability is how to control the DPF regeneration under any engine operating condition.

In general, the rising back pressure depends on the presence of the ash (as shown in Figure 7). Depth bed filtration, transition filtration, and cake filtration are three main mechanisms in the filtration process. Sappok et al. (2010a) showed that the curve of pressure drop vs. soot deposition thickness changed from the stage of depth bed filtration-soot cake filtration to the stage of soot cake filtration. This is consistent with the viewpoint of Kamp, Sappok, and Wong (2012). As illustrated in Figure 8, in a clean DPF (region a), the pressure drop rapidly incease with the ash depositing in the DPF pores (region b), corresponding to the depth bed filtration. With the continuous ash accumulation in DPF pores and filter walls, the filtration process shows transition filtration (region c). Finally, as shown in region d, ash builds a cake layer and the pressure drop gradually increases. Ash deposited in DPF occupies significantly fraction of the DPF channel volume and alters the flow conditions. Thus, ash will affect the back pressure. Nevertheless, in the further research (Sappok and Wong 2010a), it should be noted that the pressure drop sensitivity to soot loading is significantly beyond the pressure drop sensitivity to ash loading.

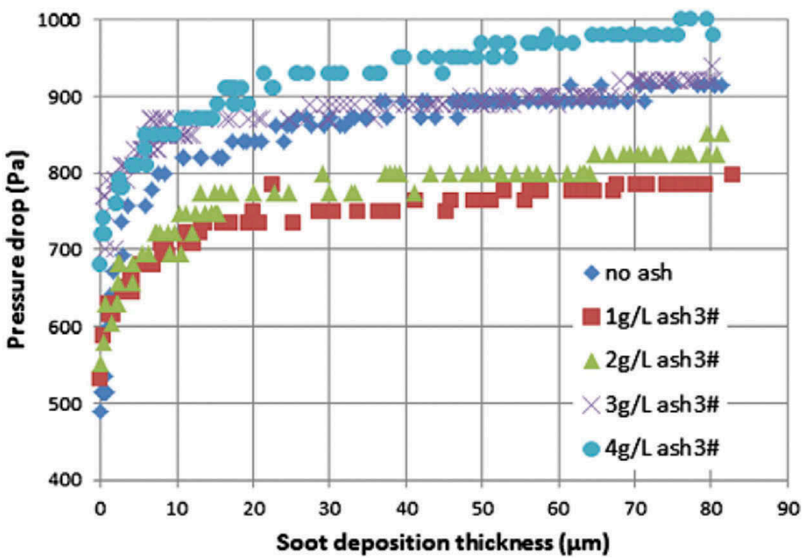

Figure 7. The effect of ash loading on pressure drop on filtration process (Fang et al. 2017).

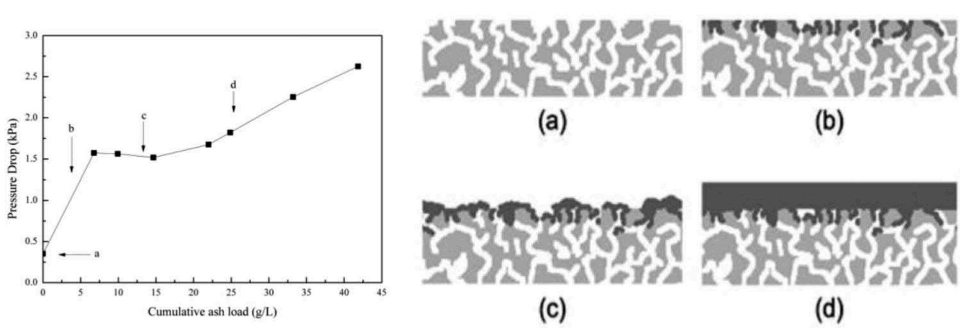

Figure 8. The DPF pressure drop curves corresponding the filtration process (Sappok and Wong 2010b).

In addition, it is worth noting that the effect of the distribution form of ash deposits in the DPF channel on the pressure drop is still debated. Gong et al. (2011) pointed out that the ash layer had a more significant influence on filter pressure drop than ash end-plugs in the research of the ash deposition styles by using a mathematical model. This is consistent with the other report (Jiang et al. 2016; Liu et al. 2015; Sappok and Wong, 2010b). On the contrary, Wang et al. (2013) considered that the sensitivity of the ash deposition styles for pressure drop depended on the properties of ash and DPF. Furthermore, they confirmed that the ash layer influence could be ignored when the average ash layer thickness is less than or equal to 11% of the clean DPF channel width. From the above-mentioned references, the debate origins from the simulation method resulting in some unwanted uncertainties and the parameters used in the simulation are usually subjective. Thus, except for making a further theoretical derivation to better understand the parameters physical meaning, related experimental work should be done by using the advanced instruments to address the dynamic process of the effects of ash forms on the pressure drop.

The presence of ash has impacts on soot oxidation characteristics. Liu et al. (2015) and Yang et al. (2013) utilized mathematical simulation and addressed that the ash accumulation increased active soot regeneration frequency and maximum regeneration temperature. The plug ash was helpful to reduce regeneration temperature and regeneration temperature gradient. Choi et al. (2015) investigated the oxidation characteristics of gasoline direct-injection) engine soot and showed that oxidation reactivity was significantly enhanced by the catalytic effects of the ash which was categorized into combustion-derived ash, unburned ash precursor, and oxidation-derived ash. The combustion-derived ash is the product of combustion of lube which has the strongest catalytic effect. The unburned ash precursor, where lube is adsorbed on, has no catalytic effect while it has weaker catalytic effect when it transfers to oxidation-derived ash that improve oxidation rates during the late oxidation process. In addition, morphology is one of the most important properties of diesel particle and the particle oxidation is closely related to it (Tan, Li, and Shen 2018; Zhang et al. 2016a). Tan and Wang (2018) investigated the effect of different ash contents on the morphology and nanostructure of diesel particulate matter and found that high ash content in particles caused a greater primary-particle diameter, the fractal dimension, and smaller fringe separation distance which could reduce the oxidation activity of diesel particle. Moreover, when it comes to CDPF performance, the ash layer can separate the soot layer and the catalyst surface, which causes the CDPF devitalization. However, Yamazaki et al. (2014) found that although the ash thickness is less than 50 μm, the catalytic oxidization still existed. They proposed "remote oxidation" mechanism that O_2^- generated on the catalyst surface first migrated to the ash surface and then to the soot particles.

As a cleaner and renewable fuel, biodiesel has attracted considerable attention. For diesel engine, biodiesel can be blended with pure diesel at any ratio and reports have demonstrated that adding biodiesel in diesel had an influence on the ash property and the soot oxidization. Liati et al. (2012b) found that the ash content (especially Ca and S compounds) and plug ash increased by adding biodiesel. Meanwhile, ash is helpful for reducing the temperature in the soot burning, while the ash layer makes the DPF regeneration deterioration with using biodiesel as fuel (Hansen, Jensen, and Jensen 2013). Although some works have been initiated to focus on the interaction between biodiesel and ash property, the knowledge about the influence of biodiesel on ash and DPF regerneration is still scarce and unlcear. Thus, some further work should be carried out in near future.

Conclusions

In order to better understand the ash deposited in DFP, a comprehensive review was presented. The relevant conclusions drawn from this work were listed as follows: (1) the ash is mainly generated from lubricating oil and the ash chemical composition are mainly Ca, Mg, P, and S. Meanwhile, the ash morphology and color are dependent on the ash chemical composition; (2) the flow-induced transport and the regeneration-reduced transport alter the mobility of the ash from the channel walls to the plug; (3) the ash cannot influence the DPF performace in the normal condition while it has important effect on back pressure and soot oxidation characteristics; (4) biodiesel blending with diesel has effect on the ash porpoises and the soot oxidization. In addition, due to the complex DPF regeneration and the unclear ash properties, there are still disagreements on the mechanism of the ash distribution process and the effect of the distribution form of ash deposits in the DPF channel on the pressure drop. In the future, the investigations on the ash physico-chemical property and the ash formation process will be helpful to address the abovementioned problems. At present, there is no limit about the sulfur, sulfate ash, and phosphorus levels in Chinese current lubricant regulations. Thus, in order to reduce the ash deposited and ensure the DPF normal operation, the more strict lubricant regulations should be implemented in China.

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