



Release of volatile organic compounds by oxidative wear of automotive friction materials

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ARTICLE INFO

Article history:

Received 9 September 2016

Received in revised form

19 November 2016

Accepted 8 December 2016

Keywords:

Wear debris

Brake pad

Organic matter

Volatile organic compounds

Polycyclic aromatic hydrocarbons

Particulate matter

ABSTRACT

Contribution of friction processes to the environmental pollution was proven in several studies, especially with respect to release of particulate emissions containing metals. Not too many researchers pay attention to volatile organic compounds formation during braking, although it is very relevant topic. When organic friction materials are used, the oxidative processes and thermal degradation always accompanies the friction processes during braking.

A low-metallic, brake lining formulation was tested using the ISO 26867 friction evaluation standard procedure in an automotive full-scale brake dynamometer. Sampling of non-airborne debris, suspended airborne particles (PM₁₀) and air was performed directly in the environmental chamber. Released volatiles and semi volatile organics were analyzed using GC/MS, Pyr/GC/MS, FTIR and carbon phase analysis. Occurrence of important groups of volatile organic compounds as PAHs and BTEXs with possible adverse effects on living organisms was confirmed.

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1. Introduction

Particulate emissions from brake wear (pad and disc) were identified among the main sources of non-exhaust emissions generated by transportation in Europe [1–3]. Frictional process occurring between pads and rotors in brakes converts kinetic energy related to the linear motion of the vehicle into thermal energy. During this process the gradual wear of the contacting components takes place which liberates brake dust [2].

The problem described above was studied by numerous researchers and addressed with an analytical or numerical simulation approaches [1–15]. It was estimated that total brake wear (evaluated for non-asbestos pads) for a small car would range between 3.2 and 8.8 mg/km depending on brake pad types [2,4,14]. 30–50 % of the brake pad mass loss was determined as the airborne emissions, 86 % of emitted particles were smaller than 10 μm (PM₁₀) and 63% of these particulates are smaller than 2.5 μm (PM_{2.5}). This suggests emission factors of 0.67–8.8 mg/km/vehicle

for PM₁₀ and 0.53–5.50 mg/km/vehicle for PM_{2.5} [2,3,5,14]. The brake dust was estimated to represent occurrence 0.3–60% of the total dust particles collected in various road sites and brake wear particles represent occurrence 16–55% of non-exhaust traffic-related PM₁₀ [3,15]. It is well-known that many hazardous compounds are released to the environment together as the brake dust [5]. Furthermore, several toxicological studies have suggested that brake wear particles can cause oxidative stress. It was suggested that not only the total mass of released wear moves importantly, but the particle quantity (number of particles released), their size, morphology and their chemical composition require further attention when their biological effects shall be addressed [3,12].

Previous studies were aimed especially to analysis of inorganic compounds released as particulate matter during braking and only a limited number of researchers focused their attention on organic compounds released from “organic” brake materials. Carbonaceous components which can be a source of released organic compounds form considerable part of brake pad formulations [3,6,8–14]. Carbon containing material, like phenolic resin, coke, graphitic carbon, nitrile rubber and aramid fibers are degraded in the friction processes due to high contact temperatures and pressures and numerous oxygenated carbonaceous compounds were observed in

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the airborne PM indicating changes in the carbonaceous components during the friction process and related oxidative wear [2–4,11]. Organic compounds can be released to the environment: i) in their gaseous form, ii) adsorbed on airborne particulate matter or iii) adsorbed on particulate matter and deposited on the brake hardware and/or the road surfaces.

The aim of this study was to determine the extent of oxidative wear by identification the organic compounds which are released to the environment during braking. The organic compounds, polycyclic aromatic hydrocarbons (PAHs) and benzene, toluene, ethylbenzene and xylenes (BTEXs) were analyzed because they are well-known hazardous substances in the environment and they were identified in brake material in our previous study [11]. The analyses were performed using gas chromatography with mass spectrometry (GC/MS), gas chromatography with mass spectrometry and connected pyrolysis unit (Pyr/GC/MS), gas chromatography with flame ionization detector (GC/FID) with the addition of carbon phase analyses and infrared spectroscopy (FTIR).

2. Experimental

2.1. Samples

A model low-metallic brake pad (containing 17% by weight of carbonaceous compounds) was used for this research.

The formulation of the model brake pad presented in Table 1 was based on compositions of commonly commercially offered brake materials. Total mass of one brake lining pad was 150 g and two pads are used in one dynamometer test. The brake lining was manufactured by mixing ingredients in Turbula multi-axis mixer (30 min), molding and hot pressing (180 °C/45,000 N/15 min) followed by post curing in air at 180 °C for 4 h [5,11].

Six samples of solid materials and ten sorption tubes (Table 2) were analyzed to identify the organic compounds: the mixture of as received brake pad ingredients (S1), the ball-milled (vibrating mill WM4 MOBIKO) model brake pad before (S2) and after (S3) the dynamometer test, the non-airborne wear debris (S4, S5) collected from the bottom of the dynamometer chamber and from the surface of pipeline conducting the cooling air into and out of the dynamometer chamber, as well as PM₁₀ particles collected on the filter situated at the air outlet (S6). The gaseous samples were collected in the sorption tubes SA500-1800 and SB500-1800.

Table 1
Formulation of the studied model brake pad composite.

| Inorganic constituents | Weight [%] | Carbon containing constituents | Weight [%] |
|--------------------------|------------|--------------------------------|------------|
| Cu containing components | 23 | Coke | 3 |
| Sn containing components | 4 | Graphite | 3 |
| Fe containing components | 25 | Aramid fibers | 1 |
| Zn containing components | 2 | Nitrile rubber | 2 |
| BaSO ₄ | 15 | Phenolic resin | 8 |
| Inorganic abrasives | 11 | – | – |
| Inorganic lubricants | 3 | – | – |

2.2. Wear test

2.2.1. Dynamometer

Brake dynamometer tests were performed using the ISO 26867:2009 and a full-scale brake automotive inertia dynamometer Link Model 2800 equipped with an environmental chamber and a wind tunnel simulating corresponding air flow [16]. It is a compact brake dynamometer controlled by the dynamometer station that performs all primary functions including selection of test parameters, control modes, display of pertinent data, monitoring of all the test functions and execution of desired test sequences. The disc brake is placed in the environmental chamber with a controlled filtered air flow capacity of 2500 m³ h⁻¹ and it consists of a ventilated rotor made of pearlitic gray cast iron (∅ 280 × 22 mm), a floating caliper FS-III of the Škoda Octavia 2003 vehicle with the piston diameter of 54 mm and two brake pads. The disc brake is connected with the inertia (65 kg m⁻²) discs that simulates the tested vehicle, and with the caliper, rotor and the torque sensor. The temperature of the rotor was recorded using K-type thermocouple positioned 1 mm below the friction surface.

The dynamometer chamber was cleaned prior to the test using pressurized water and mechanical means and, consequently, the chamber was dried and conditioned by the controlled filtered air flow of 2 500 m³ h⁻¹ for 3 h to avoid contamination from previous tests. The air at the inlet as well as at the outlet of the environmental chamber was filtered using Pocket filter KS PAK 85, the filtration class F7 (KS Klima-Service, a.s., Czech Republic), hence the surrounding air did not influence significantly the obtained results.

The internationally adopted ISO 26867:2009 is a shortened version of the European standard testing procedure AK Master [16]. It addresses sensitivity of brakes to speed, pressure and temperature as well as the common brake operation. This dynamometer test includes 24 test sequences, however, only selected sections were used for this wear study including conditions for testing of common brake operation (the sections of 500 and 1600), and for monitoring of impacts of pressure (the section of 700), speed (the section of 800) and temperature (sections of 1300 and 1800). The parameters of the individual sections are given in Table 3, including the supplementary notes about maximum achieved temperatures in the individual sections.

2.2.2. Sample collection

The non-airborne wear debris (S4) was collected by sweeping the surfaces of the dynamometer chamber and the surfaces of the wind tunnel in the outlet of the dynamometer chamber (S5) after the completion of the dynamometer test schedule. A micro-spoon shaped spatula and brushes were used for this sampling. The collected samples were placed to the tightly sealed dark vials and stored at 4 °C for several days until the analyses were performed. Each sample was well mixed, homogenized and weighted; aliquots of sufficient weights were consequently used for individual analyses.

The airborne particulate matter PM₁₀ (S6) with an aerodynamic diameter ≤ 10 μm was collected with portable sampling system MiniVol™ TAS (Airmetrics, USA). The particle size separator containing a 47 mm membrane filter Pragopor 4 (pore diameter size 0.85 ± 0.15 μm) was placed directly opposite the rotor in the dynamometer chamber in the vicinity of the outlet of the cooling air pipeline. The air from this area was drawn at 20 dm³ per min through the separator using a pump (ENVltech Bohemia, Czech Republic). The sampling was conducted over a total test period and 7.2 m³ of air was sampled in total.

The total amount of particulate matter collected on the filter was

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