



## Oxidative potential of particulate matter collected at sites with different source characteristics

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### HIGHLIGHTS

- The oxidative potential (OP) of PM was highly elevated at an underground station.
- Outdoors, PM along a highway with continuous traffic showed the highest activity.
- Contrasts in OP between sites depended on the specific OP assay used.
- The OP methods studied also differed in respect to correlation with PM composition.
- Different OP assays can provide complementary data about the oxidative properties of PM.

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### ABSTRACT

**Background:** The oxidative potential (OP) of particulate matter (PM) has been proposed as a more health relevant metric than PM mass. Different assays exist for measuring OP and little is known about how the different assays compare.

**Aim:** To assess the OP of PM collected at different site types and to evaluate differences between locations, size fractions and correlation with PM mass and PM composition for different measurement methods for OP.

**Methods:** PM<sub>2.5</sub> and PM<sub>10</sub> was sampled at 5 sites: an underground station, a farm, 2 traffic sites and an urban background site. Three a-cellular assays; dithiothreitol (OP<sup>DTT</sup>), electron spin resonance (OP<sup>ESR</sup>) and ascorbate depletion (OP<sup>AA</sup>) were used to characterize the OP of PM.

**Results:** The highest OP was observed at the underground, where OP of PM<sub>10</sub> was 30 (OP<sup>DTT</sup>) to >600 (OP<sup>ESR</sup>) times higher compared to the urban background when expressed as OP/m<sup>3</sup> and 2–40 times when expressed as OP/μg. For the outdoor sites, samples from the farm showed significantly lower OP<sup>ESR</sup> and OP<sup>AA</sup>, whereas samples from the continuous traffic site showed the highest OP for all assays. Contrasts in OP between sites were generally larger than for PM mass and were lower for OP<sup>DTT</sup> compared to OP<sup>ESR</sup> and OP<sup>AA</sup>. Furthermore, OP<sup>DTT</sup>/μg was significantly higher in PM<sub>2.5</sub> compared to PM<sub>10</sub>, whereas the reverse was the case for OP<sup>ESR</sup>. OP<sup>ESR</sup> and OP<sup>AA</sup> were highly correlated with traffic-related PM components (i.e. EC, Fe, Cu, PAHs), whereas OP<sup>DTT</sup> showed the highest correlation with PM mass and OC.

**Abbreviations:** AU, arbitrary units; AA, ascorbic acid; DMPO, 5,5-dimethylpyrroline-N-oxide; DTT, dithiothreitol; ESR, electron spin resonance; GSH, glutathione; HI, Harvard impactor; HVS, high volume sampler; MOI, micro-orifice impactor; NAQMN, National Air Quality Monitoring Network; OP, oxidative potential; PNC, particle number concentration; ROS, reactive oxygen species; RTLF, respiratory tract lining fluid; VACES, Versatile Aerosol Concentration Enrichment System.

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*Conclusions:* Contrasts in OP between sites, differences in size fractions and correlation with PM composition depended on the specific OP assay used, with OP<sup>ESR</sup> and OP<sup>AA</sup> showing the most similar results. This suggests that either OP<sup>ESR</sup> or OP<sup>AA</sup> and OP<sup>DTT</sup> can complement each other in providing information regarding the oxidative properties of PM, which can subsequently be used to study its health effects.

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## 1. Introduction

Numerous studies have shown health effects related to exposure to ambient particulate matter (PM) (Brunekreef and Holgate, 2002; Pope and Dockery, 2006). In most studies effects were linked to PM<sub>10</sub> or PM<sub>2.5</sub> mass concentrations. However, PM is a heterogeneous mixture varying in physical properties and chemical composition depending on meteorological conditions and emission sources (WHO, 2006). Current knowledge does not allow precise quantification of the health effects of individual PM components or of PM emissions from different sources (Brunekreef, 2010; WHO, 2007), although various PM characteristics, such as surface area of particles, transition metal content, surface absorbed organics components, and biological components have been proposed.

Oxidative stress has been suggested as an important underlying mechanism of action by which exposure to PM may lead to adverse health effects (Nel, 2005). Oxidative stress results when the generation of reactive oxygen species (ROS), or free radicals, exceeds the available antioxidant defenses.

The oxidative potential (OP), defined as a measure of the capacity of PM to oxidize target molecules, has been proposed as a metric that is more closely related to biological responses to PM exposures and thus could be more informative than PM mass alone (Borm et al., 2007). Oxidative potential is an attractive measure because it integrates various biologically relevant properties, including size, surface and chemical composition. Several methods for measuring OP have been developed, but no consensus has been reached yet as to which assay is most appropriate (Ayres et al., 2008). The various assays used to assess OP, each with different sensitivities to the ROS generating compounds, include Electron spin resonance (ESR) with 5,5-dimethylpyrroline-N-oxide (DMPO) as a spin trap which measures the ability of PM to induce hydroxyl radicals ( $\cdot\text{OH}$ ) in the presence of H<sub>2</sub>O<sub>2</sub> (Shi et al., 2003a,b), the ability of PM to deplete antioxidants such as ascorbic acid (AA) and glutathione (GSH) (Mudway et al., 2004), and the consumption of dithiothreitol (DTT) which is based on the ability of redox active compounds to transfer electrons from DTT to oxygen (Cho et al., 2005). In this paper, these methods will be referred to as OP<sup>ESR</sup>, OP<sup>DTT</sup>, and OP<sup>AA</sup> respectively. In addition, fluorescent-based probes have been used to quantify PM-related ROS. The most common used probe is 2,7-dichlorofluorescein (DCFH) (Landreman et al., 2008).

OP<sup>ESR</sup> and OP<sup>AA</sup> have been shown to be most sensitive to transition metals. For OP<sup>DTT</sup> typically compounds which react are organic species (e.g polycyclic aromatic hydrocarbons (PAHs) and quinones), but studies have also shown that high concentrations of transition metal ions can oxidize DTT (Charrier and Anastasio, 2012).

Only a few field studies have compared OP from different locations, with different contributing sources (Boogaard et al., 2012; Hu et al., 2008; Shi et al., 2003a). These studies have generally focused on a specific method to measure OP. Shi et al. (2003a) found that PM samples from an industrial city (Hettsted, Germany) showed 4.5 times higher OP<sup>ESR</sup> than its rural neighboring town Zerbst, despite similar PM<sub>10</sub> mass levels in the air. In the Netherlands, Boogaard et al. (2012) found that the OP<sup>ESR</sup> of PM<sub>10</sub> from major streets was 3.6 times higher than simultaneously measured PM<sub>10</sub> at urban background locations, and 6.5 times higher compared to PM<sub>10</sub> from suburban background locations. Strak et al. (2012) found only about 50% higher OP<sup>AA</sup> of PM collected at two traffic sites compared to an urban background site and no difference between the traffic sites and a farm, whereas earlier samples collected at 7 outdoor samples in the screening phase of that study

showed markedly higher OP<sup>DTT</sup> at two traffic sites compared to the other outdoor sites (Steenhof et al., 2011). Conversely, Hu et al. (2008) found rather low variability in OP<sup>DTT</sup> across six different sites in the Los Angeles area. Along with differences in spatial variation, findings from studies using different methods to measure OP also differ with regard to the most active PM fraction and relation to PM composition.

In the framework of the OPERA project (Oxidative Potential Exposure and Risk Assessment) we aim to evaluate the value of OP as a health relevant PM metric for air quality assessment and regulation. Given the limited comparative information on the different methods to measure OP the aim of the current study is to assess the OP of PM collected at different sites types and to evaluate differences between locations, size fractions and correlation with PM mass and PM composition for different measurement methods for OP.

We therefore assessed the OP from PM<sub>2.5</sub> and PM<sub>10</sub> samples collected from 5 different sites in the Netherlands, using three different measurement methods for OP (OP<sup>ESR</sup>, OP<sup>DTT</sup>, OP<sup>AA</sup>). We evaluated the correlation among the different measurement methods for OP, and compare the methods for different aspects (i.e. differences between locations, size fractions and correlation with PM mass and PM composition).

## 2. Methods

### 2.1. Study design

Within the framework of the RAPTES study (Risks of Airborne Particles—a Toxicological Epidemiological hybrid Study) PM<sub>2.5</sub> and PM<sub>10</sub> was sampled at 5 locations in the Netherlands, with different source characteristics. These locations were: an underground train station, an animal farm, a continuous traffic site (located at the exit of a tunnel of a motorway with approximately 45,000 vehicles per day) a stop&go traffic site (a major inner-city intersection with approximately 34,000 vehicles per day), and an urban background site (Strak et al., 2011). Daytime 5-hour sampling was conducted, in the period March until October 2009. On each sampling day (total 30), one site was visited and each site was visited at least 5 times.

Three a-cellular assays; dithiothreitol (OP<sup>DTT</sup>), electron spin resonance (OP<sup>ESR</sup>) of hydroxyl radical generation, and ascorbic acid depletion (OP<sup>AA</sup>) to characterize the OP of PM were selected, based on the results of a preceding intercomparison study (Yang et al., 2014). Composition of PM was measured in detail, including metals, EC/OC and PAHs.

### 2.2. Concentration measurements

Details about the air pollution measurements are described elsewhere (Strak et al., 2011, 2012). Briefly, PM<sub>2.5</sub> and PM<sub>10</sub> samples were collected using Harvard Impactors (HI) (Air Diagnostics and Engineering Inc., Naples, ME, USA). The absorbance of both the HI PM<sub>2.5</sub> and PM<sub>10</sub> filters was measured using a smoke stain reflectometer (model M43D; Diffusion Systems, London, UK) and the endotoxin content of the HI PM<sub>10</sub> samples was measured using a Limulus Amoebocyte Lisate (LAL) assay (Lonza, Basel, Switzerland). Particle number concentrations (PNC) were measured with a real-time condensation particle counter (CPC model 3022A; TSI, St Paul, MN). With a high volume sampler (model TE-6070 V equipped with TE-231 High Volume Cascade Impactor, Tisch Environmental, Clevs, OH) PM<sub>2.5-10</sub> and PM<sub>2.5</sub> samples were collected. These samples were analyzed for EC and OC using a Sunset Laboratory Thermal-Optical Carbon Aerosol Analyser, for metals

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