

Response to the comments of Anonymous Referee #1

General Comments: Tong et al. report the field measurement and laboratory experimental investigation on the production of reactive species (RS) including several radicals and hydroperoxides (ROOH) from fine PM dissolved in aqueous solutions. They explored the RS yields of fine PM from remote forest (Hyytiälä, Finland) and polluted urban air (Mainz, Germany and Beijing, China) and related these yields to different chemical constituents and reaction mechanism. They utilized ultrahigh-resolution mass spectrometry for the characterization of organic aerosol composition, electron paramagnetic resonance (EPR) spectroscopy with a spintrapping technique for the determination of the concentrations $O_2^{\bullet-}$, HO_2 , OH , carbon- or oxygen-centered organic radicals, and a fluorometric assay for the quantification of H_2O_2 concentration. They found that the mass-specific yields of radicals were lower for sampling sites with higher concentration of ambient $PM_{2.5}$ (particles with a diameter $< 2.5 \mu m$), whereas the H_2O_2 yields exhibited no clear trend. They also found that the relative fractions of different types of radicals formed by ambient $PM_{2.5}$ were comparable to the surrogate mixtures comprising transition metals, organic hydroperoxide, H_2O_2 , and humic or fulvic acids. The experiments were carefully performed, and the analyses were precise. This paper is well-written and the subject showed here are within the scope of ACP. Therefore, I would be happy to accept the manuscript once they address following issues.

Response: We thank the referee #1 for reviewing and providing positive comments. The point-by-point responses are given below. We have highlighted the changed text in blue.

Specific Comments:

In the title, abstract and throughout the text, the authors use the words “interaction of water with fine PM”. What does this “interaction” really mean? Water itself only contains H_2O , OH^- and H^+ . Although the authors wrote in page 12 “The presence of OH is related to multiple formation pathways, such as Fenton-like reactions, thermal or hydrolytic decomposition of peroxide-containing HOMs, and redox chemistry of environmentally persistent free radicals or aromatic compounds-containing humic-like substances”, it is unclear how the observed radicals are actually formed. The key issue is: they already exist and persist in PM? or they are formed just after reactions in water, e.g., $Fe^{2+} + ROOH$, or an electron-transfer reaction from OH to some components in PM? It is fruitful to clarify the meaning of “interaction” and add more discussion on the possible mechanisms of radical formation.

Response: Thank you for pointing out the unclear definition of ‘interaction’, by which we mean the chemical reactions that occur after PM is dissolved in water. For clarity, we replaced the term of ‘interaction’ with ‘aqueous-phase reactive species formation’ or ‘reactive species formation in the aqueous phase’ throughout the manuscript, including the title.

To clarify how the observed radicals are actually formed, we add the following discussion to the main text (lines 294-301):

Environmentally persistent free radicals (EPFR), are known to pre-exist in PM_{2.5} at mass-specific concentration levels of ~0.2 to ~2 pmol μg⁻¹, which are an order of magnitude higher than the typical mass-specific aqueous-phase radical yields of ~0.02 to ~0.2 pmol μg⁻¹ (Arangio et al., 2016;Vejerano et al., 2018;Tong et al., 2019;Chen et al., 2020). While some EPFR may be water-insoluble (Chen et al., 2018), others may directly contribute to the C-centered and O-centered radicals trapped by BMPO or participate in redox reactions yielding •OH and O₂• radicals (Khachatryan et al., 2011;Arangio et al., 2016). The latter have such short chemical lifetimes that they have to be formed upon dissolution of the investigated samples immediately prior to trapping by BMPO.

More discussions on the radical formation mechanism are given in the responses below.

The authors infer that OH comes from HOMs containing peroxide-groups. However, it has been reported that the Fe²⁺ + ROOH (R=CH₃, C₂H₅) reaction in water produces RO + OH⁻, rather than RO⁻ + OH [Chevallier et al., Atmos. Environ. 2004, 38, 921]. RO would produce HO₂. Then, what is a major source of OH-radicals in the present system?

Response: Thank you for raising this important point. To explain the PM source-dependent formation mechanisms of aqueous-phase radicals, we add the following discussions in the main text (lines 282-293):

We speculate that hydrolytic or thermal decomposition of ROOH may play a major role in the formation of RS by PM_{2.5} from remote forest locations like Hyytiälä, where large fractions of peroxide-containing HOM have been detected (Mutzel et al., 2015;Tröstl et al., 2016;Tong et al., 2019;Pye et al., 2019;Roldin et al., 2019;Bianchi et al., 2019). ROOH can generate •OH and O-centered organic radicals through decomposition (ROOH → RO• + •OH) and Fenton-like reactions (Fe²⁺ + ROOH → Fe³⁺ + RO• + OH⁻; Fe²⁺ + ROOH → Fe³⁺ + RO⁻ + OH•) (Tong

et al., 2016). Interconversion of RO•, R• and ROO• radicals can lead to the formation of O₂⁻ and H₂O₂ (Chevallier et al., 2004; Tong et al., 2018), which can further react with Fe²⁺ to form •OH (Fe²⁺ + H₂O₂ → Fe³⁺ + •OH + OH⁻). In PM_{2.5} from urban areas, transition metal ions and HULIS are expected to play a major role in aqueous-phase formation and interconversion of •OH, O₂⁻ and H₂O₂ (Lloyd et al., 1997; Valavanidis et al., 2000; Zheng et al., 2013; Hayyan et al., 2016; Lakey et al., 2016; Tan et al., 2016; Kuang et al., 2017; Ma et al., 2018; Li et al., 2019).

A PAM chamber experiment would produce atmospherically irrelevant RS species [Peng and Jimenez, Chem. Soc. Rev., 2020, 49, 2570]. A major RS of PM in ambient air would be a species possessing multiple -OOH formed via autoxidation process involving intramolecular H-abstractions (i.e., HOM). Thus, the high vs. low O₃/OH condition critically influence the product identities and the distributions. Does this concentration gap influence the current conclusion? The authors should comment on the issue.

Response: Thank you for this valuable comment. We agree that there is an O₃/•OH concentration gap from the laboratory chamber experiment to the real ambient air, and the distributions and identities of HOM in SOA depend on the absolute concentrations of O₃/OH and the ratio of oxidant to precursor concentration. However, it is not possible for us to resolve the issue in this study and we plan to accommodate the problem in future studies. To clarify this point, we add the following text in the SI (lines 46-50):

To note, there is a gap between the concentration of gas phase O₃ or •OH in laboratory chamber experiment and ambient air. In the PAM chamber, one might form atmospherically irrelevant RS under the high oxidant conditions (Peng and Jimenez, 2020). The distributions and identities of HOM in SOA depend on the absolute concentrations of O₃ or •OH and also the concentration ratio of oxidant to precursor, which warrants further analysis.

Can the authors estimate how much O₂ is dissolved in solutions? The presence of O₂ in solutions could induce autoxidation reactions that give OH-radical signals of EPR [Floyd and Wiseman, Biochim. Biophys. Acta, 1979, 586, 196]. O₂ in solutions could also influence the Fenton-like chemistry and HO_x cycles in the system [Chevallier et al., Atmos. Environ. 2004, 38, 921]. If the authors perform degassing of solutions (e.g., by Argon) before measurement, are the same results obtained? It would be better to perform such a test to understand the mechanism in more detail.

Response: Our measurements were conducted under ambient conditions and a degassing was not performed. We assume that the concentration of oxygen in the aqueous phase (~0.29 mM) is determined by Henry's law and constant over reaction time. To identify the influence of aqueous-phase O₂ on the radical formation by SOA in water, we also determined the radical yields of β-pinene SOA in degassed water (with ultrapure N₂ for ~1 h and keep the N₂ exposure during the extraction operation). Indeed, we found that dissolved O₂ increases the radical yield of 1 mM SOA by ~20%. In both experiments, the majority of detected radicals were •OH (RF > 80%). To clarify the potential influence of aqueous-phase O₂ on the radical formation by SOA, the following sentences were added to the SI (lines 122-133):

Aqueous-phase O₂ has been suggested to be capable of inducing autooxidation reactions and influencing the Fenton-like chemistry as well as HO_x cycles of organic hydroperoxides in water (Floyd and Wiseman, 1979;Chevallier et al., 2004). We thus compared the radical yields of β-pinene SOA in non-degassed and degassed water (with ultrapure N₂ for ~1 h and keep the N₂ exposure during the extraction operation). We found that OH radicals are always the major species trapped by BMPO and detected with EPR within both environments. Moreover, ~20% more radicals were observed upon dissolving β-pinene SOA in non-degassed water (1 mM), reflecting the important role of O₂ in the radical formation by SOA in water. These experimental results can be explained by our recent modelling analysis, which shows that absence of O₂ will lead to the recombination of C-centered radicals R• and interrupt formation of peroxy radicals and superoxide radicals from R• (Tong et al., 2017;Tong et al., 2018). In this study, measurements were conducted under ambient, non-degassed conditions. We assume that the concentration of oxygen in the aqueous phase (~0.29 mM) is determined by Henry's law and remain constant over reaction time.

C-centered radicals are expected to rapidly react with O₂ or recombine in condensed phases. Why were they so abundant (Figures 2 and 4)? What types of C-centered radicals can be assumed? The authors should comment on the issue.

Response: To explain the observation of substantial amounts of C-centered radicals, we add the following discussions in the main text (lines 265-271):

The high yield of C-centered radicals can be explained by rapid trapping of C-centered organic radicals (R^\bullet) by BMPO in the liquid phase (De Araujo et al., 2006). In the aqueous extracts, we applied a large excess of BMPO (10 mM of BMPO vs. $\sim 1 \mu\text{M}$ of trapped radicals), and the estimated pseudo-first-order rate coefficient for R^\bullet reacting with BMPO ($9 \times 10^5 \text{ s}^{-1}$, (Tong et al., 2018)) is much higher than the estimated R^\bullet recombination rate coefficient (2.4×10^3 , (Simic et al., 1969; Tong et al., 2018)). Moreover, rearrangement reactions in water can convert RO^\bullet into R^\bullet (Chevallier et al., 2004), which may warrant further investigation.

BMPO-OOH must decompose into other products in solutions (it decreases as a function time in Fig. S6). What are the decomposition products? Do the products influence the RS yields? The authors should comment on the issue.

Response: The referee is correct, there are multiple sinks of the BMPO-OOH, including the conversion of BMPO-OOH to BMPO-OH, direct dissociation, and reactions with other reactants (e.g., metal ions and radicals) in the aqueous extracts of PM (Tong et al., 2018). Due to the general limitation of the spin trapping and EPR spectrometry techniques in differentiating different source radicals (e.g., primary versus secondary), we cannot assess the influence of decomposition products of BMPO-radical adducts quantitatively without further studies. To clarify this point, we add the following text in the SI (lines 116-120):

To note, there are multiple sinks of the BMPO-radical adducts, which include the conversion of BMPO-OOH to BMPO-OH, direct dissociation, and reactions with other reactants (e.g., metal ions and radicals) in the aqueous extracts of PM (Tong et al., 2018). It remains a challenge to assess the influence of decomposition products of BMPO-radical adducts on the radical detection in this study, warranting further studies.

Fig. 3 shows H_2O_2 is the dominant RS in $\text{PM}_{2.5}$, while radical species contribute just as minor components. What is the main source of the observed high H_2O_2 ? Furthermore, from the viewpoint of adverse health effects of PM inhalation, if $[\text{H}_2\text{O}_2] \gg [\text{radicals}]$ in lung epithelium lining fluid, how important are these radicals? It would be better to add some discussion on these issues.

Response: To note, ROS concentrations in lung epithelium lining fluid (ELF) may be different compared to those observed in water because of the presence of enzymes and antioxidants in the

ELF (Tong et al., 2018). The H₂O₂ formation by ambient PM has been suggested to be strongly associated with α -hydroxyhydroperoxides, transition metals, and quinones (Arellanes et al., 2006; Charrier et al., 2014; Lakey et al., 2016; Wei et al., 2021). The two-step reduction of O₂ with reductive substances (e.g., transition metals and semiquinone, SQ, radicals) to form superoxide radicals (SQ/TM^Z + O₂ → Q/TM^{Z+1} + O₂^{•-}) and subsequently H₂O₂ (SQ/TM^Z + O₂^{•-} → Q/TM^{Z+1} + H₂O₂) may be the major source of the observed high H₂O₂ in this study. Excessive amounts of exogenous H₂O₂ not only deplete enzymes and antioxidants, but also act as precursor of [•]OH, inducing further respiratory damages (Halliwell et al., 2000). Indeed, compared to H₂O₂, the measured radical concentrations are relatively low, however, the radicals are much more reactive. Moreover, the EPR method may not detect all radicals produced but rather a fraction that is trapped with BMPO before undergoing other radical termination reactions. Therefore, the overall health effect of cumulatively formed number of radicals is unclear and warrants follow-up studies. We add the following text in in lines 322-326 to discuss the source of aqueous-phase H₂O₂ in PM_{2.5} extracts:

The strong increase of H₂O₂ with increasing PM_{2.5} concentration is consistent with earlier studies identifying a wide range of redox-active organic and inorganic aerosol components that can produce H₂O₂ in the aqueous phase (Gunz and Hoffmann, 1990; Anastasio et al., 1994; Zuo and Deng, 1997; Arellanes et al., 2006; Chung et al., 2006; Hua et al., 2008; Möller, 2009; Wang et al., 2010; Wang et al., 2012; Anglada et al., 2015; Herrmann et al., 2015; Lakey et al., 2016; Tong et al., 2018; Bianco et al., 2020).

Minor Comment:

Line 858, “participated”

Response: Thanks. We corrected the typo.

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