

We thank their reviewer for the thoughtful and constructive comments, and we have revised the manuscript accordingly. Listed below are our point-to-point responses (in **blue**) to the comments (in **black**) and changes of the manuscript (in **red**).

Responses to Reviewer 2:

Review comments on the manuscript "Photoaging of Phenolic Secondary Organic Aerosol in the Aqueous Phase: Evolution of Chemical and Optical Properties and Effects of Oxidants" in EGU sphere. The manuscript addresses the long-term aqueous aging of aqSOA formed from the photooxidation of guaiacyl acetone (GA) by OH radicals or the photosensitizer model compound 3,4-dimethoxy-benzaldehyde (DMB) using Pyrex tube experiments and performing positive matrix factorization (PMF) analysis of combined HR-AMS and UV-vis spectral data.

Questions and remarks:

1. Could the authors elaborate more the description of the experimental and analytical method, as it is a bit vague at the moment. It is not clear to the reader if the experiment is performed in a closed system or if it is, open to the atmosphere, if oxygen is present or not? Could the authors describe the potential contribution of singlet oxygen to the conversion within the system when the photosensitizer dimethoxybenzaldehyde and oxygen are present?

The Pyrex tubes containing the reaction solutions were capped but not airtight during the experiment. To ensure homogeneity, the solution was stirred continuously. In addition, the cap was briefly removed when samples were collected for instrumental analyses. Thus, oxygen should have been present in the solution throughout the reaction, allowing singlet oxygen ($^1\text{O}_2^*$) to form via energy transfer from $^3\text{C}^*$ to ground state O_2 and act as an oxidant for the phenolic precursor.

However, in our previous studies on the reactions of phenols with $^3\text{DMB}^*$ under similar conditions, $^1\text{O}_2^*$ has been shown to be an insignificant oxidant for the phenols compared to the rapid $^3\text{DMB}^*$ -mediated reactions (Smith et al., 2014). In that work, we observed that phenols decay faster under acidic conditions, which is opposite to the fact that the reactions of $^1\text{O}_2^*$ with deprotonated phenolate ions are much more rapid (Canonica et al., 1995; Tratnyek and Hoigne, 1991). This observation suggests that $^3\text{C}^*$ is the major oxidant for phenols in the reaction, while $^1\text{O}_2^*$ is a minor contributor.

In response to these comments, we have added the following sentences to Sec 2.1 of the updated manuscript.

Line 114: During the photoreaction, the solutions were continuously stirred. The Pyrex tubes were capped but not hermetically sealed, and the caps were briefly removed during sample collection. Due to the presence of oxygen in the reaction system, secondary reactive oxygen species (ROS) such as singlet oxygen ($^1\text{O}_2^*$), superoxide/hydroperoxyl radicals ($\text{O}_2^{\bullet-}/\text{HO}_2^{\bullet}$) and $\bullet\text{OH}$ can be generated in the solution via energy transfer from $^3\text{C}^*$ to dissolved O_2 (Vione et al., 2014; Zepp et al., 1977), electron transfer from the intramolecular charge-transfer complex of DMB ($\text{DMB}^{\bullet+/\bullet-}$) to O_2 (Dalrymple et al., 2010; Li et al., 2022), and the reactions between DMB ketyl radical and

O₂ (Anastasio et al., 1997). However, according to our previous studies (Smith et al., 2014), singlet oxygen is expected to contribute only minimally to the oxidation of GA in this reaction system. In addition, the negligible loss of GA and DMB in the dark controls suggests there was negligible evaporation of the precursor or the photosensitizer during the experiments.

2. Could the guaiacylacetone itself act as a photosensitizer?

Compared with DMB, GA absorbs much less sunlight due to its lower extent of conjugation. This suggests that GA is not an effective photosensitizer, and the formation of triplet excited state of GA is negligible in the reaction. In a study by Smith et al. (2016), it was reported that GA is significantly less light-absorbing compared to other phenolic and non-phenolic carbonyl photosensitizers, and shows negligible direct photolysis or oxidation with syringol. To show the light absorptivity difference between GA and DMB, we have updated Figure S5 to include the MAC spectra of both compounds.

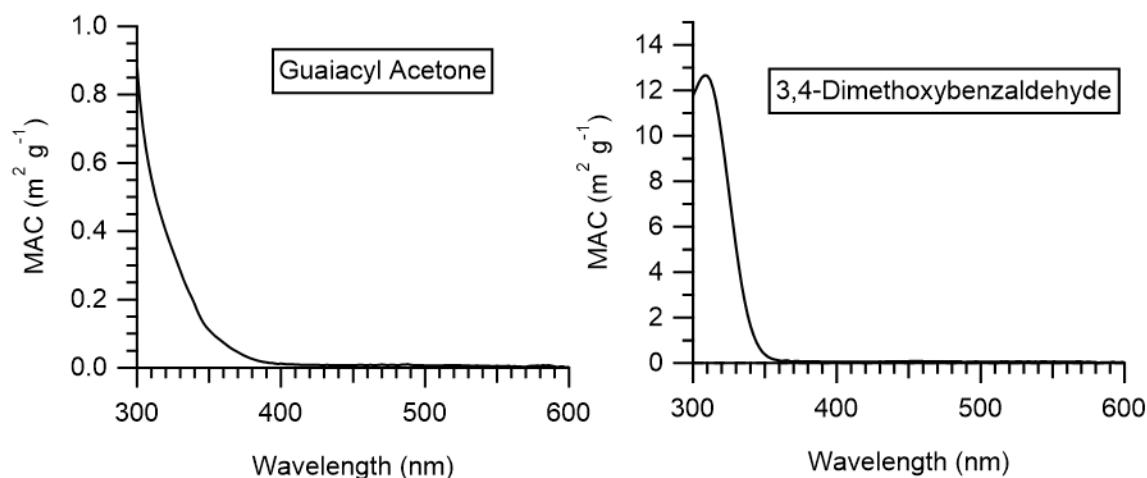
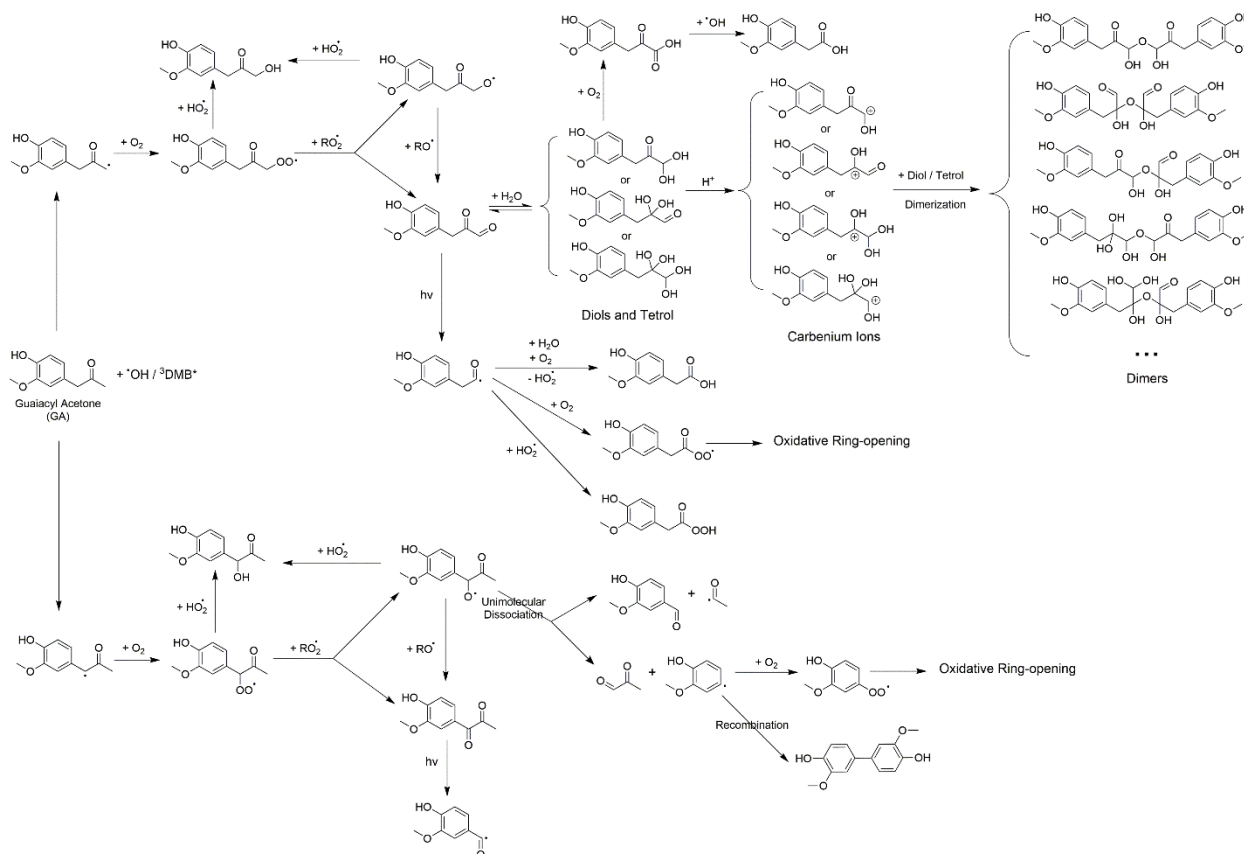


Figure S5. Mass absorption coefficient (MAC) spectra of guaiacyl acetone (GA) and 3,4-dimethoxybenzaldehyde (DMB)

3. Would the authors expect a similar oligomer formation yield and conversion rate if 4-propylguaiacol had been used?

We would expect oligomer formation from the photoreaction of 4-propylguaiacol with ³C*/•OH since it is a methoxyphenol. However, different phenols can exhibit significantly different oligomer formation rate and yields. For example, Yu et al. (2014) reported that the aqSOA formed from guaiacol shows a significantly higher mass fraction of oligomers than that from phenol. In addition, the formation rates of oligomers from the coupling of phenoxy radicals can be affected by steric hindrance by large functional groups (Steenken and Neta, 2003).

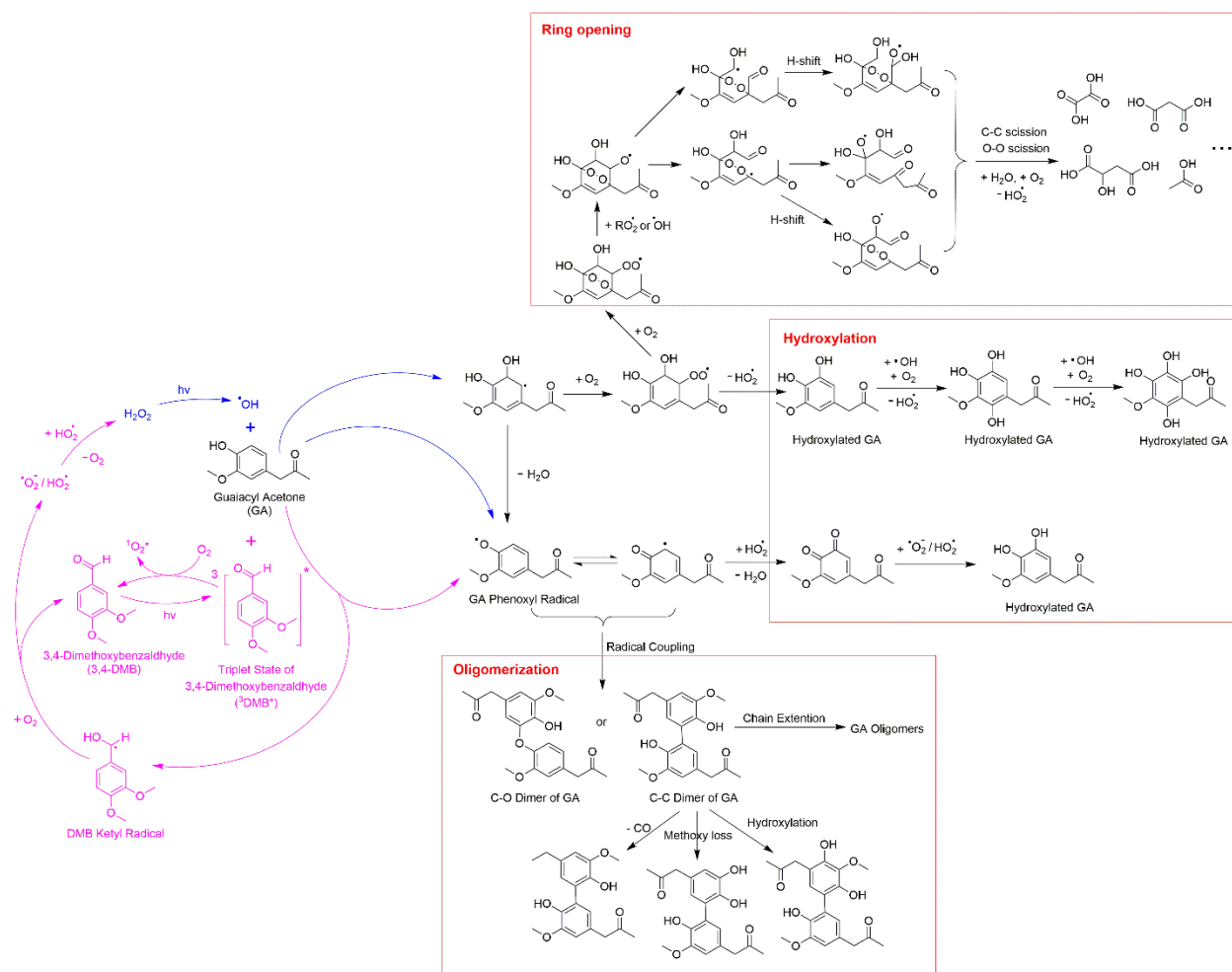
Moreover, it is important to note that GA has a ketone functional group which is absent in 4-propylguaiacol, and the reactions triggered by the ketone group can also contribute the production of oligomers as shown in Scheme 2 in the updated manuscript.



Scheme 2. Postulated reaction pathways triggered by the ketone functional group of GA.

4. What would be the general reaction mechanism in the photosensitizer system in the presence of GA and DMB after the first step of H-atom abstraction or electron transfer? Is addition of the photosensitizer possible?

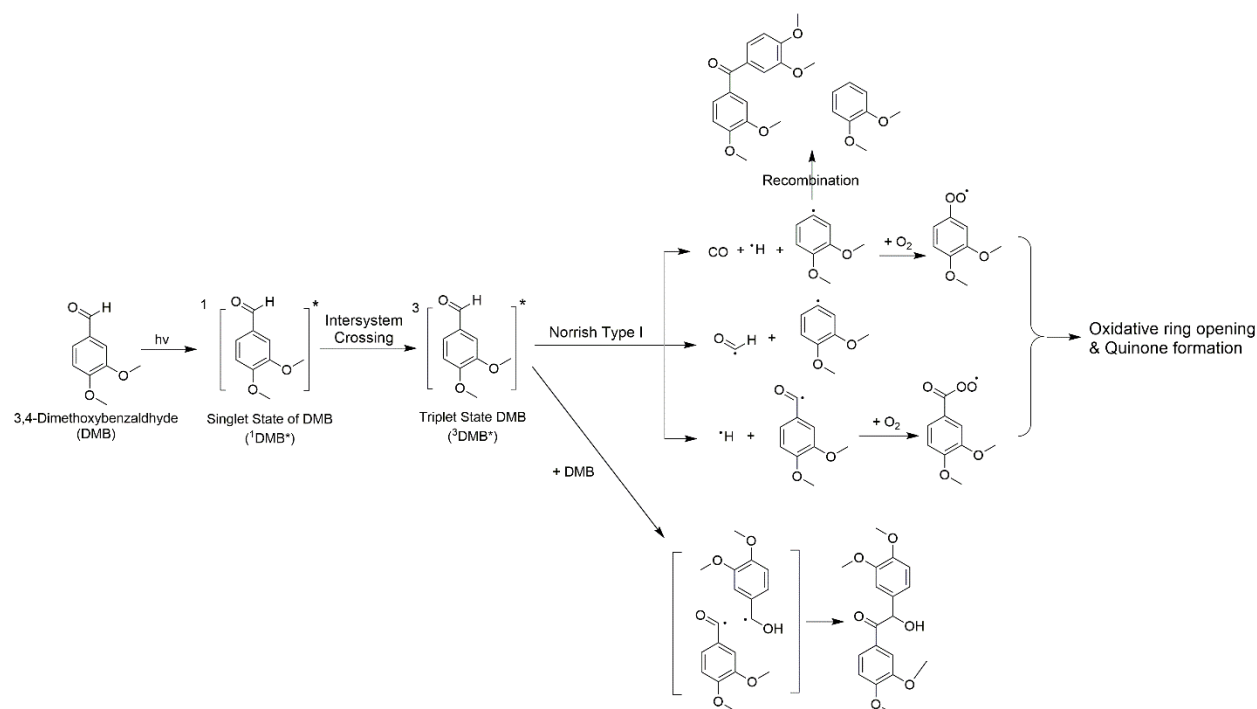
The reaction of $^3\text{DMB}^*$ with GA can lead to H-atom abstraction/electron transfer at the phenol functional group, generating a GA phenoxyl radical and a DMB ketyl radical. The GA phenoxyl radicals can undergo coupling to form dimers and higher oligomers (Mabato et al., 2023; Yu et al., 2014). Alternatively, the GA phenoxyl radical can also react with $\text{HO}_2^*/\bullet\text{O}_2^-$ in the solution to produce o-quinone, which is subsequently converted to hydroxylated GA products (D'Alessandro et al., 2000; Steenken and Neta, 2003). The DMB ketyl radical can be reduced by O_2 to form $\text{HO}_2^*/\bullet\text{O}_2^-$ and regenerate DMB (Anastasio et al., 1997). Scheme 1 has been added to the updated manuscript to illustrate the reaction pathways.



Scheme 1. Postulated reaction pathways triggered by the phenol functional group of GA.

On the other hand, during the photoexcitation, DMB may dissociate via Norrish reactions to generate phenyl and benzoyl radicals (Shen and Fang, 2011) which can undergo recombination to produce dimeric products and dimethoxybenzene (Dubtsov et al., 2006; Theodoropoulou et al., 2020). Therefore, as this reviewer suggested, DMB may participate in the reactions as a reactant. Scheme S1 has been added to the supplementary to illustrate potential DMB-participated reaction pathways.

However, considering the low concentration of DMB (20 times lower than GA) and its slow decay rate observed in our reaction system, it is unlikely that the reactions involving DMB played a significant role, and their contribution to the aqSOA is expected to be insignificant.



Scheme S1. Postulated reaction pathways for the photodegradation of 3,4-dimethoxybenzaldehyde. The mechanisms are adapted from previous studies on benzaldehydes (Berger et al., 1973; Dubtsov et al., 2006; Shen and Fang, 2011; Theodoropoulou et al., 2020).

5. What would be the difference in the first oxidation products formed by the photosensitizers compared to OH radicals?

It is important to note that based on our AMS measurements, the aqSOA was found to be a mixture of different products from the beginning, reflecting the competition of different reaction pathways (e.g., oligomerization, functionalization, and fragmentation) throughout the aqSOA formation and aging. The postulated reaction pathways are shown in Schemes 1 and 2.

Based on our PMF analysis, both the 1st-generation aqSOA factor (which is enrich with oligomers) and the 2nd-generation factor (which is enriched with functionalized GA monomers) were found to build up upon irradiation in both the $\bullet\text{OH}$ and the ${}^3\text{C}^*$ reactions. The mass spectral profiles of the 1st-generation aqSOA factors from the ${}^3\text{C}^*$ and $\bullet\text{OH}$ reactions were found to be highly similar, suggesting similar compositions. However, the mass fraction of the 1st-generation factor in the ${}^3\text{C}^*$ -aqSOA is higher than that in the $\bullet\text{OH}$ -aqSOA initially. For example, when 20% of GA was consumed, the 1st-generation factor accounted for $\sim 75\%$ of the total $\bullet\text{OH}$ -aqSOA mass (Figure 6e), while it constituted 90% of the total ${}^3\text{C}^*$ -aqSOA mass (Figure 7e). This suggests that oligomerization plays a more important role in the early stage of ${}^3\text{C}^*$ -mediated reactions as compared to $\bullet\text{OH}$ reactions. A more detailed comparison between the initially formed ${}^3\text{C}^*$ -aqSOA and $\bullet\text{OH}$ -aqSOA is shown in Sec 3.1 of the manuscript.

6. What would be the result of the involvement of oxygen? Would the resulting peroxy radicals lead to the formation of oligomers, what is the authors' opinion? If not, could this explain the lower yield of oligomers in OH radical-induced oxidation? How likely is alkyl/phenoxy-like radical recombination in the presence of oxygen at the steady-state concentrations used?

The presence of oxygen may influence the reactions in several ways, as described below.

- 1) In the •OH-mediated reaction, the dihydroxycyclohexadienyl radical generated via OH addition can further react with O₂ to form a peroxy radical (Scheme 1). The peroxy radical can eliminate a HO₂• to produce hydroxylated products. Alternatively, the peroxy radical can also cyclize to generate bicyclic radicals (Dong et al., 2021; Suh et al., 2003), which can undergo ring cleavage pathways to produce fragmented products such as small carboxylic acids, aldehydes, and ketones.

However, we do not expect dimerization of the peroxy radicals to be significant in our reaction system since we did not observe the formation of organic peroxide (ROOR). Oligomer formation in this study is likely primarily through the coupling of phenoxy radicals, which is more enhanced in the ³C*-mediated reaction. Therefore, we agree with this reviewer that negligible dimerization of peroxy radical may also partly explain the lower yield of oligomers in the •OH-mediated reaction.

In summary, the presence of O₂ in the •OH reaction can enhance ring-cleavage reactions, while the oligomerization of phenoxy radicals can be suppressed (Dong et al., 2021). However, we cannot determine the degree to which the presence of O₂ inhibits oligomerization.

- 2) In the ³C*-mediated reaction, the energy transfer from ³DMB* to ground state O₂ produces singlet oxygen (¹O₂*; Scheme 1). The ¹O₂* reacts with phenols mainly through 1,4-cycloaddition route to produce quinone products (Al-Nu'airat et al., 2019; García, 1994). A higher O₂ concentration increases the importance of ¹O₂* pathways, thus promoting quinone formation. However, it also suppresses the formation of phenoxy radicals and the dimerization of phenoxy radicals.

However, as discussed in our response to the first Question of this reviewer, the ¹O₂* reaction is expected to be a minor contributor to GA oxidation in this study.

- 3) Furthermore, in the ³C* reaction, DMB ketyl radical can react with O₂ to produce superoxide/hydroperoxy radical (O₂•-/HO₂•) which can further react to form H₂O₂ and •OH (Scheme 1). The O₂•-/HO₂• and •OH play an important role in the hydroxylation and ring-opening reactions of GA. Therefore, the presence of O₂ in the ³C* reaction can contribute to more promoted hydroxylation and ring-opening pathways.
- 4) The addition of ground state O₂ to the phenoxy radical is possible, which leads to the formation of a phenoxy peroxy radical (Batiha et al., 2012; Steenken and Neta, 2003). However, this reaction is very slow and does not appear to significantly affect the fate of phenoxy radicals (Batiha et al., 2012). Therefore, the effect of this pathway on the dimerization of phenoxy radicals is expected to be negligible.

To demonstrate the aqueous-phase reaction pathways of GA and the role of O₂ in the reactions, we have added “Section 3.2 Aqueous-phase Reaction Pathways of Guaiacyl Acetone” and Schemes 1 and 2 to the updated manuscript.

7. In the authors' opinion, what are the main oxidation products of the OH radical reaction with GA?

The composition of the aqSOA evolves over time, with oligomers and functionalized products being more important in the early stages of the reaction. However, as the aging process continues, ring-opening products (e.g., small acids, aldehydes and ketones) become more dominant. These findings are demonstrated in Figures 6e, 6g, and 6i, and are consistent with the proposed reaction mechanisms. We have added Schemes 1 and 2 to the updated manuscript to illustrate the major reaction pathways and the formation of these products.

8. On page 7, line 166, it is mentioned that the products formed can evaporate more easily. Later (line 177), the formation of carboxylic acids and compounds formed by the degradation of aromatic rings is mentioned. How likely is it that these compounds will evaporate?

In this study, we did not characterize the volatility of the products. However, our previous study (Jiang et al., 2021) observed the formation of five ring-opening carboxylic acids, including formic acid, acetic acid, malonic acid, malic acid, and oxalic acid, from the aqueous photoreactions of GA. As shown in the following table, most of the detected acids are volatile and are expected to evaporate readily from the aerosol phase.

Name	Formula	K_H ($M \text{ atm}^{-1}$) (EPI Suite, HENRYWIN)	Vapor Pressure at 298 K (Pa) (EPI Suite, MPBPWIN)	Fraction sorbed to airborne particles (EPI Suite, AEROWIN, Mackay Model)
Formic Acid	HCHO	1.96E+03	4.78E+03	4.23E-08
Acetic Acid	C2H4O2	3.40E+03	2.29E+03	1.15E-07
Malonic Acid	C3H4O4	2.46E+08	1.34E-01	9.78E-05
Malic Acid	C4H6O5	1.19E+09	3.90E-04	8.31E-01
Oxalic Acid	C2H2O4	4.15E+07	7.46E-01	1.34E-03

9. Could the authors indicate how long the H₂O₂ (100 μ M) as well as the DMB is present in the solution before it decays by the photochemistry? How many times is DMB involved in a reaction as a photosensitizer before it is degraded?

Based on the fact that GA follows pseudo-first-order decay until completely consumed in both the \bullet OH and the $^3\text{C}^*$ reactions, we deduce that the concentrations of H₂O₂ and DMB stayed relatively constant during the initial aqSOA formation period (i.e., 24 hours for H₂O₂ and 3.5 hours for DMB). After GA is consumed, we were not able to monitor the concentration of H₂O₂ but we measured the concentration changes of DMB using HPLC-DAD and the results are shown in Figure 1i.

The times that a DMB molecule can be involved in the reactions as a photosensitizer are influenced by several factors, such as the rate of DMB photoexcitation, the quantum yield of $^3\text{DMB}^*$, the rate of H-atom abstraction/electron transfer between $^3\text{DMB}^*$ and GA, other potential sinks for $^3\text{DMB}^*$ (e.g., O₂) in the system, and the rate of DMB photo-dissociation. Although this is an interesting question, it is beyond the scope of this work, which focuses on understanding the chemical and optical properties of the GA aqSOA.

10. Is it possible that GA acts as a photosensitizer in the GA + H₂O₂ system used and reacts with H₂O₂, which is subsequently more important than the production of OH radicals by H₂O₂ photolysis, since the absorption of GA is somewhat greater compared to H₂O₂ in the specific wavelength range?

Based on our previous study, we do not expect GA to be an efficient photosensitizer, as both direct photolysis of GA and ³C* formation from GA (monitored by the reaction between syringol and GA) were found to be negligible under a similar condition (Smith et al., 2016).

11. Did the authors do any experiments with GA in the absence of an oxidant?

Our previous study has reported that GA exhibits negligible direct photolysis under simulated solar irradiation (Smith et al., 2016). The following sentence has been added to the manuscript.

Line 162: Based on a previous study (Smith et al., 2016), direct photolysis of GA is expected to be negligible in this study.

12. How justified here is the statement (on page 10 line 215) that the steady-state concentration of OH is the same or similar?

We assumed a steady-state concentration of •OH during the initial 24 hours of illumination based on the fact that GA followed pseudo-first-order decay during that time period. However, as GA was depleted after 24 h of irradiation, we were unable to predict •OH concentration based on the decay of GA. It is possible that the concentration of •OH decreased due to the consumption of H₂O₂, especially towards the end of the prolonged aging (72 hr).

13. The end of section 3.4 is difficult to follow and is full of speculation. Could the authors sharpen the end of the section for clarity and with concrete numbers, e.g., how likely evaporation is? This brings me back to the experimental description, where it is not clear whether evaporation may or may not play a role in this study, so the experimental design is not well described within the manuscript.

We would like to point out that AMS only measures the low volatility products, while the volatile and semi-volatile products evaporated during the aerosolization and drying. For clarification, the following sentences have been added to Sec 2.2.

Line 129: The liquid samples were atomized in argon (Ar, industrial grade, 99.997 %) followed by diffusion drying (Jiang et al., 2021). This process allowed volatile and semi-volatile products to evaporate, leaving only the low-volatility products in the particle phase, which were characterized by AMS.

In addition, the following text has been added to Sec 2.1 to better describe the experimental method.

Line 114: During the photoreaction, the solutions were continuously stirred. The Pyrex tubes were capped but not hermetically sealed, and the caps were briefly removed during sample collection. Due to the presence of oxygen in the reaction system, secondary reactive oxygen species (ROS) such as singlet oxygen (¹O₂*), superoxide/hydroperoxyl radicals (O₂•⁻/HO₂•) and •OH can be generated in the solution via energy transfer from ³C* to dissolved O₂ (Vione et al., 2014; Zepp et

al., 1977), electron transfer from the intramolecular charge-transfer complex of DMB (DMB^{•+/-}) to O₂ (Dalrymple et al., 2010; Li et al., 2022), and the reactions between DMB ketyl radical and O₂ (Anastasio et al., 1997). However, according to our previous studies (Smith et al., 2014), singlet oxygen is expected to contribute only minimally to the oxidation of GA in this reaction system. In addition, the negligible loss of GA and DMB in the dark controls suggests there was negligible evaporation of the precursor or the photosensitizer during the experiments.

To better interpret the effects of additional oxidants on the aqSOA aging, the following text has been added to Sec 3.4.

Line 386: Likewise, the addition of extra •OH or ³C* results in more extensive mass loss of the •OH-aqSOA, with reductions of 88% or 79% of the aqSOA mass observed at the end of the photoaging, respectively. These levels of mass loss were significantly higher than no extra oxidant (i.e., 62%). These findings suggest that the presence of additional •OH or ³C* accelerates the photochemical aging process and leads to increased formation of volatile and semi-volatile products that subsequently evaporate.

In summary, this manuscript, which certainly has its merits and is quite interesting in its present form, might be improved more in terms of the clarity and coherence of its scientific basis to enable a recommendation for acceptance.

We have carefully considered this comment, as well as all the other comments, and have revised our manuscript accordingly. Specifically, we have made changes to address the concerns raised by the reviewer, including modifying the language in certain sections and providing additional clarification and explanation where needed. We have also added new data and reaction schematics to support our findings. We believe that these revisions have strengthened our manuscript and improved its overall quality.

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