

Response to the comments of Anonymous Referee #2

[Atmospheric Chemistry and Physics, MS ID: acp-2016-1081]

Title: Mixing state of oxalic acid containing particles in the rural area of Pearl River Delta, China: implication for seasonal formation mechanism of Secondary Organic Aerosol (SOA)

General comments:

The authors present a comprehensive analysis of the mixing state of oxalic acid in the Pearl River Delta area of China using a single particle aerosol mass spectrometer (SPAMS). The topic of the paper is important with regard to understanding formation pathways of secondary organic aerosol (SOA). The main findings are the following: oxalic acid containing particles accounted for <3% of total particles; in summer heavy metals containing particles were the largest group containing oxalic acid while in winter it was the biomass burning group; the majority of oxalic acid particles were internally mixed with sulfate and nitrate; the fraction of oxalic acid particles containing ammonium increases significantly in winter versus summer. A couple of interesting speculations are made about acid-catalyzed reactions and Fenton like reactions. The paper is written well. The title and abstract are mostly reflective of the contents of the paper. The results are quite interesting and I support publication after each of my comments below are adequately addressed.

Response: Thank you for your comments. These comments are all valuable and very helpful for revising and improving our paper, as well as the important guiding significance to our researches. We have studied these comments carefully and have made corrections. Our responses to the comments are itemized below.

Anything about our paper, please feel free to contact me at limei2007@163.com

Best regards!

Sincerely yours

Mei Li

April 6, 2017

Specific comments and point by point responses:

1. While the title of the paper and parts of the manuscript make reference to comparison of summer and winter and also mention the word 'seasonal', it is important to consider that the authors are only looking at two short-term periods spanning 2 weeks. I suggest a relaxation of words in parts of the paper that make it seem as though full seasonal coverage was obtained. At the minimum, 'seasonal' needs to be revised in my view for the title.

Response: As suggested by you and other reviewers, we have removed "seasonal" from the title, and the title "[Mixing state of oxalic acid containing particles in the rural area of Pearl River Delta, China: implication for seasonal formation mechanism of Secondary Organic Aerosol \(SOA\)](#)" has been changed to "[Mixing state of oxalic acid containing particles in the rural area of Pearl River Delta, China: implications for the formation mechanism of oxalic acid](#)".

2. Line 189: These percentages are quite interesting to me and they seem low based on how ubiquitous the literature suggests oxalic acid is in particles. Can the authors comment more about how these percentages compare to previous reports?

Response: Yang et al. (2009) has measured the oxalic acid-containing particles in the urban area of Shanghai by ATOFMS and found 15,789 oxalate-containing particles, accounting for 3.4% of the total collected particles. In this work 13,109 and 20,504 of oxalic acid-containing particles were obtained in summer and winter separately, accounting for 2.5% and 2.7% of the total detected particles. The abundance of oxalic acid-containing particles in this work was lower than the reported studies in the urban area of Shanghai (3.4%), which was possibly due to less anthropogenic precursors for oxalic acid at the rural sampling site in Heshan. Higher abundance of oxalic acid particles (1-40%) was observed in the much cleaner western Pacific Ocean by Sullivan et al. (2007), which corresponded to higher ambient concentration of DCAs ($19 \pm 4.8\%$) in total particulate organic matter. From the reported studies in PRD (Yao et al. 2004; Ho et al. 2011), the abundance of DCAs was 1-3.5% in total organic matter, which was much lower than those in the western Pacific Ocean, leading to lower percentage of oxalic acid-containing particles in this work. We have added the comparison between this work and the study in Shanghai in the manuscript. Content of "[The percentage of oxalic acid-containing particles in total particles in this work was comparable to the reported value in the urban area of Shanghai \(3.4%\) \(Yang et al., 2009\). However, these percentages are in general much lower than those reported in cleaner environments such as the western Pacific Ocean where oxalic acid was found in up to 1-40% of total particles due to little anthropogenic influences \(Sullivan and Prather, 2007a\).](#)" has been added in lines 192-198.

3. Line 189-193: While the authors refer readers to the Supplement, it would be useful here to at least provide a little more detail as to how the categorization was done assuming that not all readers will go to the Supplement. The categories are very

important for the results, so some more discussion is warranted here as to how this was done.

Response: We have added the classification rules for oxalic acid particles in the manuscript and explain the differences among the EC, OC, ECOC type, and several literatures are cited in the manuscript to support these classification rules. “All oxalic acid particles are classified into eight types: elemental carbon (EC), organic carbon (OC), elemental and organic carbon (ECOC), biomass burning (BB), heavy metal (HM), secondary (Sec), sodium-potassium (NaK) and dust.” has been changed to “The oxalic acid containing particles are classified into eight types in the following order: elemental carbon (EC), organic carbon (OC), elemental and organic carbon (ECOC), biomass burning (BB), heavy metal (HM), secondary (Sec), sodium-potassium (NaK) and dust. Different type particles are identified according to characteristic ion markers and dominant chemical species (Table S1): (1) particles containing abundant carbon clusters like $\pm 12[\text{C}]^{+/-}$, $\pm 24[\text{C}_2]^{+/-}$, $\pm 36[\text{C}_3]^{+/-}$ with relative peak area more than 0.5% are classified as EC type, (2) any remaining particles containing abundant signals of $27[\text{C}_2\text{H}_3]^+$, $43[\text{C}_2\text{H}_3\text{O}]^+$ and hydrocarbon clusters with relative peak area more than 0.5% are classified as OC type, (3) any remaining particles containing signals of $\pm 12[\text{C}]^{+/-}$, $\pm 24[\text{C}_2]^{+/-}$, $37[\text{C}_3\text{H}]^+$ and $43[\text{C}_2\text{H}_3\text{O}]^+$ with relative peak area more than 0.5% are classified as ECOC type, (4) any remaining particles containing abundant signals of $39[\text{K}]^+$ (peak area > 1500) with relative peak area of $-59[\text{C}_2\text{H}_3\text{O}_2]^-$ and $-73[\text{C}_3\text{H}_5\text{O}_2]^-$ simultaneously more than 0.5% are classified as BB type, (5) any remaining particles containing signals of $55[\text{Mn}]^+$, $56[\text{Fe}]^+$, $63/65[\text{Cu}]^+$, $64[\text{Zn}]^+$ and $208[\text{Pb}]^+$ with relative peak area more than 0.5% are classified as HM type, (6) any remaining particles containing abundant signals of $18[\text{NH}_4]^+$ (peak area > 50), $-62[\text{NO}_3]^-$ (peak area > 100) and $-97[\text{HSO}_4]^-$ (peak area > 100) are classified as Sec type, (7) any remaining particles containing abundant signals of $23[\text{Na}]^+$ (peak area > 1500) and related species are classified as NaK type, (8) any remaining particles containing signals of $40[\text{Ca}]^+$, $56[\text{CaO}]^+$ and related species are classified as dust type. The rules for oxalic acid particles classification in the current work have been reported in previous studies (Sullivan and Prather, 2007b; Yang et al., 2009; Zhang et al., 2013; Li et al., 2014).” in lines 199-221.

4. Line 289-291: Could the lower mixing height in the winter have contributed in some way to this finding (i.e. more stagnant aerosol and perhaps more aged)?

Response: The wind speed was from 0.3 to 4.0 m s^{-1} with an average of 1.6 m s^{-1} during the sampling period in winter, which indicated a rather stagnant atmospheric condition. The stagnant atmospheric condition in winter was favorable for the aging process of aerosols and might have contributed to the broader size distribution of oxalic acid particles. Unfortunately the height of boundary layer was not available in this work. Nevertheless, the relative abundance of oxalic acid particles had no obvious increase at night and the lower mixing height may not be an important factor during the formation process of oxalic acid in the winter. We will further examine their relationships in our next study.

5. Line 337-338: The wording here in this sentence and the general paragraph appear to be too strong in my view since the authors did not unambiguously prove that photo-Fenton reactions are even occurring. Aren't these just speculations? I suggest to use less strong language and to differentiate better between proved findings and speculations.

Response: Indeed these speculations and proposed results should be addressed in a proper expression. “Although •OH production from Fenton reactions can both occur under dark and light radiation conditions, only photo-Fenton reactions had significant contribution to the enhancement of oxalic acid particles in the current work.” has been changed to “Although •OH production from Fenton reactions can both occur under dark and light radiation conditions, photo-Fenton reactions may have more contribution to the enhancement of oxalic acid particles in the current work.” in lines 369-371.

6. Line 329-359: have the authors considered all other factors that could affect the diurnal behavior of oxalic acid particles, such as meteorological factors or different emissions types during the day. Can the authors comment on how the profiles of other species look in Figure 7 if they were plotted such as sulfate, EC, nitrate? Is rush hour traffic influential at all in any of the discrepancies between the peak of ozone and the other parameters currently shown in Figure 7? While these other factors may not be important, it is still important to mention that various other potential factors were considered.

Response: The diurnal variations of meteorological factors such as temperature, RH and wind speed are shown in Figure S7.

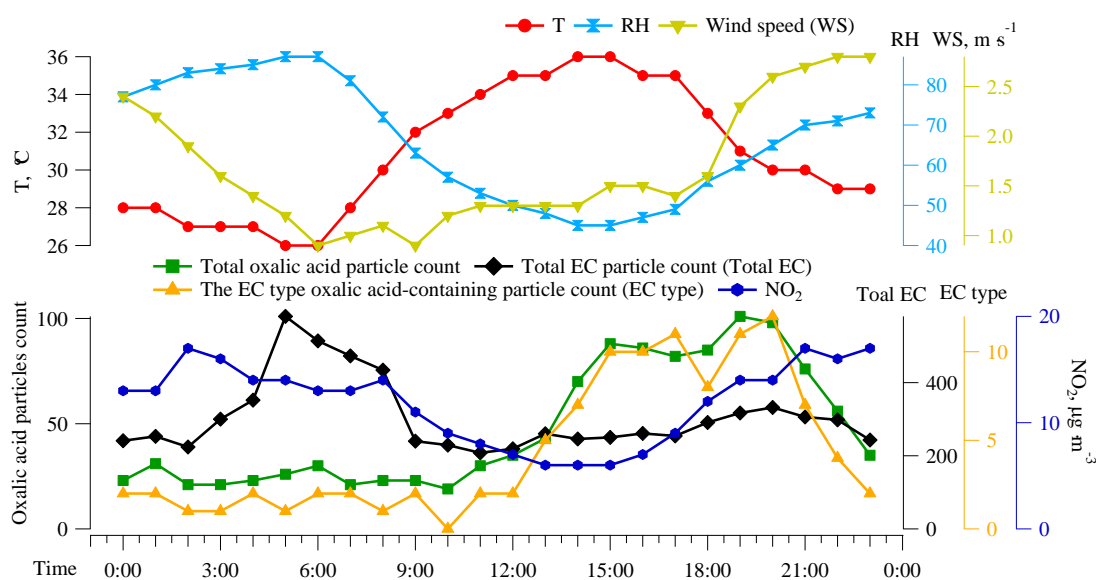


Figure S7. The diurnal variations of temperature (T), RH, wind speed (WS), oxalic acid particles, total EC particles, the EC type oxalic acid-containing particles and ambient NO₂ concentrations from July 28 to August 1 in 2014.

The high temperature between 9:00 and 19:00 was favorable to the secondary

processing of organic precursors. The wind speed was low during the whole day, especially between 9:00 and 18:00, which provided a stagnant environment for the increase in oxalic acid after produced from photochemical process. The impacts of RH and traffic emissions on the formation of oxalic acid have been illustrated Figure S5 and S7, respectively. The influence from traffic emission was investigated through the diurnal variations of total EC type particles and NO₂ (Figure S7). The EC type particles increased from 12:00 to 21:00, which had same variation as total oxalic acid, but NO₂ followed the rush hour pattern with two peaks from 5:00 to 8:00 and from 18:00 to 21:00. Traffic emission is not expected to have a large contribution to oxalic acid in this study. Oxalic acid was found to be internally mixed with sulfate and nitrate with mixing ratio of 93% and 94% in summer, and the sulfate and nitrate type oxalic acid particles were not classified in this work, so the diurnal patterns of sulfate and nitrate are not presented in Figure S7.

We have added the related discussion in the manuscript. “The diurnal patterns of temperature, wind speed are presented in Figure S7. The high temperature between 9:00 and 19:00 was favorable to the secondary processing of organic precursors. The wind speed was low during the whole day, especially between 9:00 and 18:00, which provided a stagnant environment for the increase in oxalic acid produced from photochemical process. The influence from traffic emission was investigated through the diurnal variations of total EC type particles and NO₂ (Figure S7). The EC type particles increased from 12:00 to 21:00, which had same variation as total oxalic acid, but NO₂ followed the rush hour pattern with two peaks from 5:00 to 8:00 and from 18:00 to 21:00. Traffic emission is not expected to have a large contribution to oxalic acid in this study.” has been added in lines 417-426 in the manuscript.

7. Section 3.3: the discussion and analysis surrounding the acid-catalyzed hypothesis is too thin in my view. Were such relationships not observed in the summer, and if not, then why? Could another reasonable explanation be that that precursors of oxalic acid and the organosulfate species are co-emitted? To strengthen this conclusion, the discussion and analysis needs to be more convincing with also more discussion of other relevant words using field data to point to this mechanism of acid-catalyzed formation of SOA.

Response: In summer few organosulfate containing particles were observed and hence we have only discussed the relationship between the oxalic acid and organosulfate containing particles in winter. The temporal trend of organosulfate-containing oxalic acid particles in winter has been added in Figure S8, which exhibited a similar pattern as the total oxalic acid particles. The percentage of organosulfate-containing oxalic acid particles in total oxalic acid particles ranged from 0 to 16.4% with the highest ratio observed on February 8. The linear regression between oxalic acid particles and organosulfate particles in Figure 9b has been replaced by the correlation between organosulfate-containing oxalic acid particles and total oxalic acid particles, and the robust correlation ($r^2=0.81$) between them supports a possible production of oxalic acid from acid-catalyzed reactions.

We agree with more evidence and discussion are very helpful to support the

proposed mechanism connecting oxalic acid formation and aqueous phase chemistry at acidic conditions, and following discussions as well as a new Figure 8 have been added in lines 428-455:

“Despite lower O_3 concentrations and photochemical activity in winter, oxalic acid particles were still prevalent in carbonaceous particles, especially BB type particles. While oxalic acid was found to be internally mixed with sulfate and nitrate both in summer and winter, the nitric acid was only observed in oxalic acid particles in winter, indicating a strongly acidic nature of oxalic acid particles in winter. Considering a possible connection of oxalic acid production with the acidic environment, the temporal concentrations of oxalic acid, sulfate and nitrate were investigated through their peak areas in the carbonaceous type oxalic acid particles including EC, OC, ECOC and BB type in Figure 8. The peaks of m/z -62[NO_3^-] and -97[HSO_4^-] represent nitrate and sulfate, respectively. Nitrate, sulfate and oxalic acid showed very similar variation patterns in winter, indicating a close connection of their co-existence. Although nitric acid was found in the oxalic acid particles, the acidity of the oxalic acid particles was not estimated since the real-time concentration of inorganic ions was not available during the sampling period in winter. Instead the relative acidity ratio (R_{ra}), defined as the ratio of total peak areas of nitrate and sulfate to the peak area of ammonium (m/z 18[NH_4^+]), was used (Denkenberger et al., 2007; Pratt et al., 2009). The R_{ra} of carbonaceous type oxalic acid particles ranged from 7 to 114 with an average value of 25 (Figure 8), indicating an intensely acidic environment of carbonaceous type oxalic acid particles in winter. Several studies have reported potential production of oxalic acid from acid-catalyzed aqueous phase reactions in aerosols (Carlton et al., 2006; Carlton et al., 2007; Tan et al., 2009). In this work the acidic environment of the carbonaceous type oxalic acid particles and similar variation patterns among oxalic acid, sulfate and nitrate may suggest a relationship between the degradation of organic precursors and the acidic chemical process. However, the temporal change of R_{ra} did not follow a similar trend as the peak area of oxalic acid in most particles, possibly due to the multi-step formation of oxalic acid influenced by many factors such as precursors, liquid water content and ion strength (Carlton et al., 2007; Cheng et al., 2013; Cheng et al., 2015).”

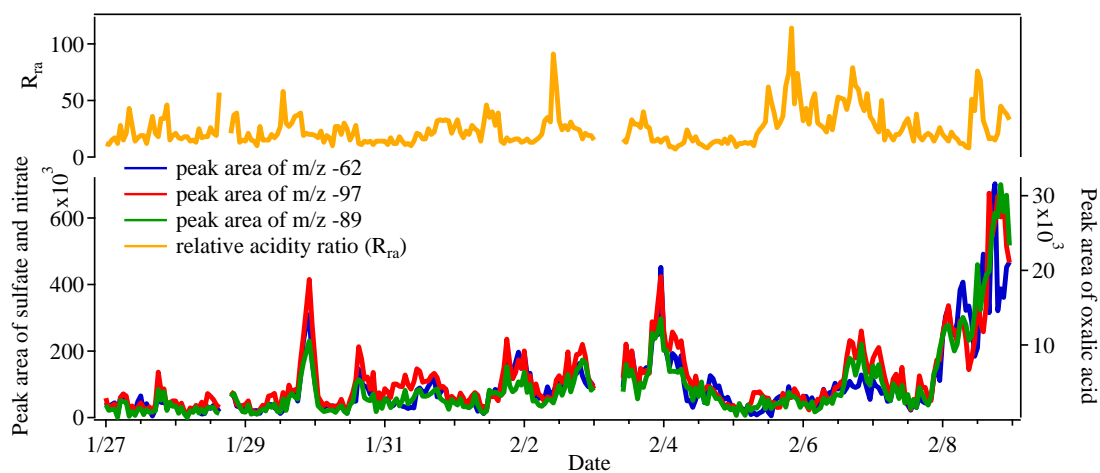


Figure 8. The temporal variations of peak area of nitrate, sulfate and oxalic acid, and the relative acidity ratio (R_{ra}) in carbonaceous type oxalic acid particles in winter.

The related discussions about Figure 9 and Figure S8 have been revised: “The organosulfate derived from glyoxal requires acidic aqueous environment of particles, and herein is used as an indicator of acid-catalyzed ageing process of organic compounds. The temporal variation of organosulfate ($m/z=-155$) containing particles during the entire sampling period in Heshan, China is shown in Figure S6. During the episode, oxalic acid particles had moderate linear correlation with organosulfate particles (Figure 9b).” has been changed to “The formation of organosulfates from glyoxal requires an acidic aqueous environment, which can be used as an indicator of acid-catalyzed ageing process of organic compounds. The temporal trend of organosulfate-containing oxalic acid particles in winter is shown in Figure S8, which exhibited a similar pattern as the total oxalic acid particles during the whole sampling period in winter. The percentage of organosulfate-containing oxalic acid particles in total oxalic acid particles ranged from 0 to 16.4% with the highest ratio observed in the episode (February 8). The linear regression between total oxalic acid particles and organosulfate-containing oxalic acid particles in the episode is exhibited in Figure 9b, and the robust correlation ($r^2=0.81$) between them suggests that oxalic acid and organosulfate may share similar formation process.” in lines 476-486.

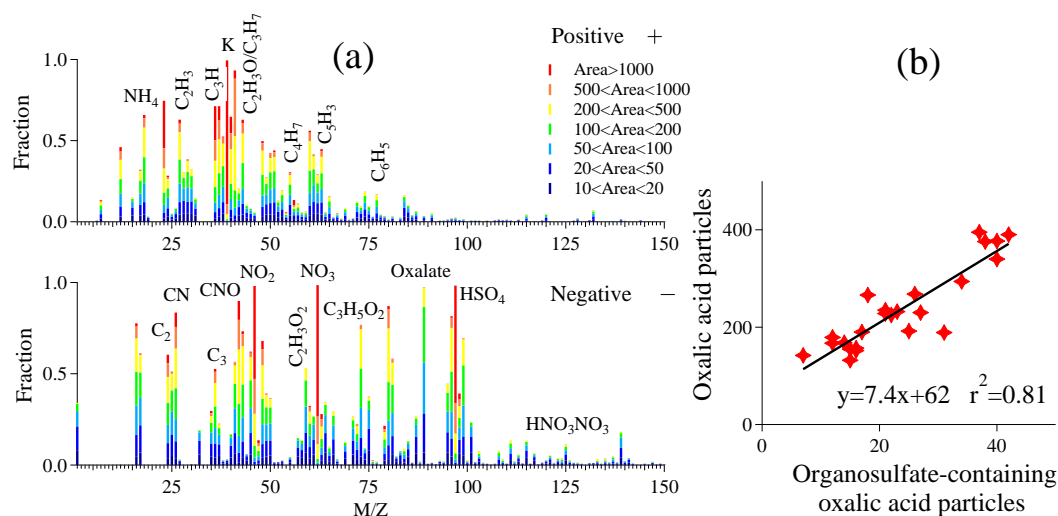


Figure 9. The comprehensive study of oxalic acid particles increase on Feb 8, 2015: (a) The digitized positive and negative ion mass spectrum of oxalic acid particles during the episode; (b) Linear regression between total oxalic acid particles and organosulfate-containing oxalic acid particles (m/z -155).

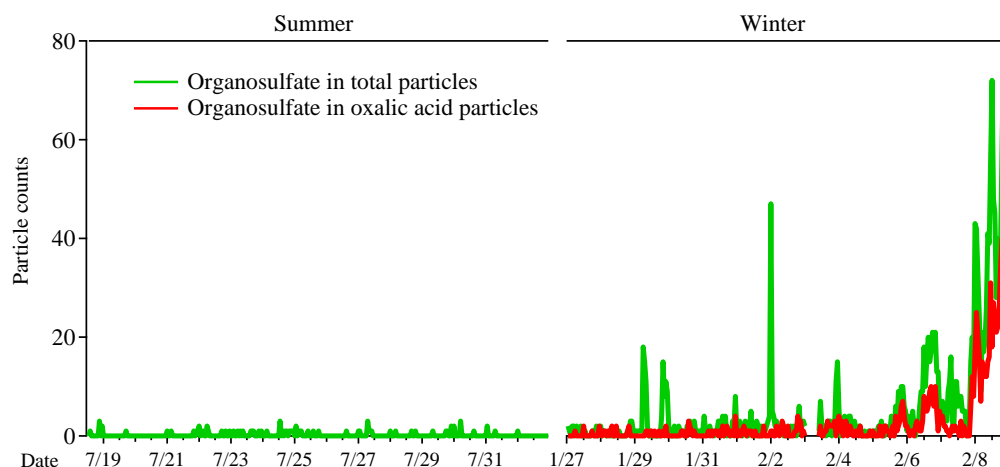


Figure S8. Temporal variation of organosulfate ($m/z=-155$) containing particles in total particles and in oxalic acid particles in Heshan, China.

“The temporal trend of organosulfate-containing oxalic acid particles in winter is also shown in Figure S8, which exhibited a similar pattern as the total oxalic acid particles. The percentage of organosulfate-containing oxalic acid particles in total oxalic acid particles ranged from 0 to 16.4% with the highest ratio observed on February 8.” has been added in the supplement material.

8. Line 107-110: This line needs a revision because it is not entirely fair. It should be noted somewhere around this section that quite a bit of work has been done with fast time resolution on aircraft to address the issue of meteorological uncertainty and temporal resolution limitations. These various studies have discussed the formation pathways leading to oxalic acid with detailed in-cloud and out-of-cloud measurements:

Wonaschuetz, A., et al. (2012). Aerosol and gas re-distribution by shallow cumulus clouds: an investigation using airborne measurements, *J. Geophys. Res.*, 117, D17202, doi:10.1029/2012JD018089.

Sorooshian, A., et al. (2006). , *J. Geophys. Res.* 111, D23S45, doi:10.1029/2005JD006880.

Sorooshian, A., et al. (2007). Particulate organic acids and overall water-soluble aerosol composition measurements from the 2006 Gulf of Mexico Atmospheric Composition and Climate Study (GoMACCS), *J. Geophys. Res.*, 112, D13201, doi:10.1029/2007JD008537.

Response: We have revised these sentences, and these references related to in-cloud and aqueous phase formation of oxalic acid have also been cited in the manuscript. “Several studies have found a tight correlation between oxalic acid and sulfate in ambient particles, implying that aqueous chemistry leads to the formation of oxalic acid in aerosols and cloud droplets (Yao et al., 2002; Yao et al., 2003; Yu et al., 2005; Sorooshian et al., 2007a; Miyazaki et al., 2009).” has been changed to “A number of ground based and airborne field studies have found a tight correlation between oxalic acid and sulfate in ambient particles and cloud droplets, relating

aqueous phase chemistry to the formation of oxalic acid in aerosols and cloud droplets (Yao et al., 2002; Yao et al., 2003; Yu et al., 2005; Sorooshian et al., 2006; Sorooshian et al., 2007a; Sorooshian et al., 2007b; Miyazaki et al., 2009; Wonaschuetz et al., 2012; Wang et al., 2016).” in line 99-104. “However, the exact formation pathways of oxalic acid in ambient particles are still unknown due to the complexity of meteorological condition and the temporal resolution limitations of conventional filter sampling studies and bulk chemical analysis.” has been changed to “However, the detailed formation mechanisms of oxalic acid from photochemistry and aqueous phase chemistry in ambient aerosols are still not comprehensively understood and need to be further studied.” in lines 111-113.

9. Figures: Font size needs to increase in many of the figures for labels.

Response: We have checked all the Figures and Tables and revised the font size in most figures in the manuscript and the supplement material.

References:

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