

acp-2020-1009: Seasonal analysis of submicron aerosol in Old Delhi using high resolution aerosol mass spectrometry: Chemical characterisation, source apportionment and new marker identification

Response to the reviewers' comments

We thank the reviewer for providing detailed and helpful comments on our manuscript. Below we respond to each reviewer comment in turn (reviewers' comments are shown in italics), indicating the changes we have made.

Response to Anonymous Referee #2

General Comments: The manuscript by Cash et al. describes a chemical characterization and source apportionment work using high resolution aerosol mass spectrometry (HR-AMS) data collected in one of the most polluted and populated areas in Old Delhi, India in 2018. It covered 3 time periods: pre-monsoon (around 1 month), monsoon (around 15 days), and post-monsoon (around 6 weeks). The study identified two traffic-related factors (NHOA and HOA), two burning-related factors (SFOA and SVBBOA), a cooking factor (COA), and two SOA factors (LVOOA and SVOOA) using positive matrix factorization (PMF). In the end, the Authors suggest developing air quality policies by mitigating emissions from crop residue burning, open waste burning, and traffic activities. Cash et al. were able to reveal the mystery of high chloride concentration using HR-AMS, which has strong links with the burning sources, especially in the post-monsoon period. In addition, with the rather well-resolved PMF solution, it is certainly a valuable reference for AMS users in the future (e.g., the mass spectra of NHOA, SFOA, SVBBOA). Collecting and analyzing such a dataset from a high temperature, high relative humidity environment remains challenging, but the authors were able to justify their results using both independent and internal tracers. The multilinear regression analysis is a useful tool for this dataset considering there is no reference HR mass spectrum available for a unique environment, like old Delhi. I believe the contribution of this work is significant and meets the scope of the ACP. Overall, the English are perfect, but the whole manuscript is not so well-organized and wordy, which makes it difficult to follow. For this reason, I will suggest accepting this manuscript only after re-organizing of the text and considering some major issues listed below.

Major issues: Section 3.3. focus too much on the technical details in justifying the PMF factors using some independent or internal tracers, which is important but not the scope of this study. Thus, I will only summarize it within few paragraphs, and move some figures and texts into SI. I suggest focusing more on the discussions of each source you retrieved from PMF. The separation between section 3 and 4 and the long text makes the storyline discrete and difficult to follow. In addition, it also makes figures often not the closest to the texts that explain the

figures, which makes it difficult to read. Please condense the captions you have in this manuscript, especially for the figures in the SI.

We have removed parts of the text in the results section that on further consideration we felt were less relevant and we have moved other parts to the SI. Some figures (Figures 12(a) and 13) that are not key to the storyline have also been moved into the SI (Now Figures S13 and S14). We thank the reviewer for these suggestions as they have improved the transition from the results to the discussion section.

In response to the reviewer's comment on this matter we have altered and reduced the majority of the figure captions. However, we are strongly of the opinion that they should retain sufficient information to permit figures to be understandable to the reader without reading information from the main text.

Technical comment: Have you tried to run the bootstrap analysis to see if you get a rather robust result? If it is just a single PMF run, I would argue you might suffer from rotational ambiguity. How did you cope with that? Have you ever tried to use factor profiles from “7f_ac_SI_C1” as a-prior information to run PMF but with rather loose constraints?

We did conduct bootstrapping analysis but, the results showed variability in the solutions. The variation calculated through bootstrapping analysis includes mainly random error with partial contribution from rotational ambiguity (Brown et al., 2015). Little rotational ambiguity was found when using FPEAKS, which means the variation found from bootstrapping is most likely related to random error. However, the solutions were found to contain a high degree of correlation between factors meaning that bootstrapping analysis may be incorrect or ambiguous for this dataset (Ulbrich, 2011).

The bootstrapping resampling method could be potentially altered in order to solve this issue. The resampling method used by the PET tool (used in this study) resamples by randomly replacing subsets of original rows (mass spectra) with other rows from the original matrix (Ulbrich, 2011). The EPA PMF software however goes one step further and uses a resampling method called block bootstrapping where a new dataset is created from randomly selecting non-overlapping time periods or ‘blocks’ (i.e. of length 3-days) (Paatero et al., 2014). This is likely a better method for this dataset and it may help to reduce the effects of serial correlation (or correlation between factors). It is however unlikely to account for large variations in factors between different time periods. Factors such as SVBBOA, for example, increase from 0 to 40 $\mu\text{g m}^{-3}$ going from pre-monsoon to post-monsoon. After resampling, ‘blocks’ from the pre-monsoon will be placed next to ‘blocks’ from the post-monsoon and this will create a large amount of variation that is not due to model error or solution instability. Previous studies have described similar issues and have used their own resampling method (Hemann et al., 2009).

It may be possible to apply bootstrapping analysis on a seasonal basis to counteract these issues, but this is beyond the scope of this study. There is also the time series dependence on the boundary layer which is unlikely to be accounted for using standard resampling methods. Additionally, it is computationally demanding and software, such as EPA PMF, do not support the analysis of a 2.39673×10^7 data point matrix.

Using SEED analysis, we determined that there is variance in the solution and to deal with this potential ambiguity we applied multilinear regression analysis on possible SEEDS which is a well establish method (Allan et al., 2010; Young et al., 2015; Elser et al., 2016; Reyes-Villegas et al., 2016). This method deals with time series dependencies as the external tracers are similarly affected by the same meteorological effects such as the boundary layer.

We have not tried to use the profiles as a priori information to run in ME-2 but unless we constrain each factor differently, we fail to see how this would change the result from using variations in the SEED. If factors were to be constrained differently to one another, there are no previous high-resolution studies in Delhi on which to base this. We are therefore strongly of the opinion that applying no constraints to novel PMF profiles provides a more objective result.

Detailed comments:

1. Line 73: Add (Lalchandani et al., 2021; Tobler et al., 2020) to your citation.

These citations have been added: “A growing number of studies in Delhi, and other locations in India, have reported large concentrations of chloride, especially during the morning hours at ~7-9 a.m. (Sudheer et al., 2014; Chakraborty et al., 2018; Gani et al., 2019; Acharja et al., 2020; Reyes-Villegas et al., 2020; Tobler et al., 2020; Lalchandani et al., 2021).”

2. Finger 1: Combine Fig1, Fig.2, and Fig 5 to give a better overview of the chemical composition as well as the data availability.

We thank the reviewer for this suggestion and have combined the three figures to create a better summary of the data (new Figure 1).

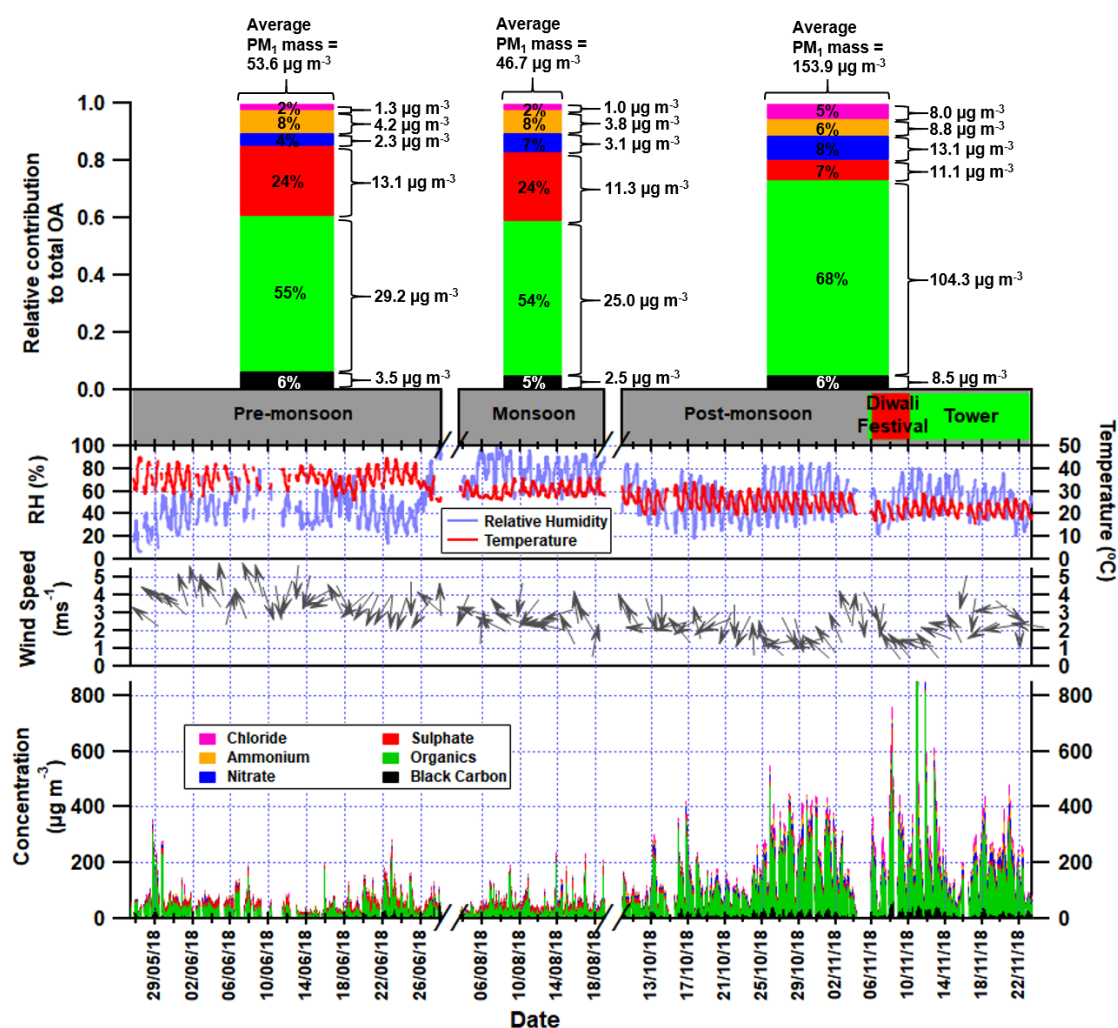


Figure 1 – First Panel: Average relative contributions of chloride, ammonium, nitrate, sulphate, organic aerosol and black carbon to the total PM_{10} mass loadings in the pre-monsoon, monsoon and post-monsoon periods. The average concentrations of each species are shown to the right of each bar (see Table S5 for values and statistics). Second panel: Gantt chart showing the measurement periods where the red region shows the Diwali festival and the green region shows when the inlet was moved to a 30 m tower. Third panel: time series of the relative humidity and the temperature for the three measurement periods. Fourth panel: time series of the wind speed with arrows showing wind direction. Fifth panel: time series of stacked concentrations of aerosol species showing total PM_{10} .

3. Line 129-131: Provide RF, RIEs somewhere, also mention how many times you have calibrated the instrument. Details on the calibration results are summarised in Table S2 and the number of times the HR-AMS was calibrated have been added to the text: “The HR-TOF-AMS was calibrated fortnightly over the three campaigns (11 calibrations in total) for its ionisation efficiency of nitrate (IE) and the relative ionisation efficiency (RIE)...”

Table S2 – Relative ionisation efficiencies (RIE), ionisation efficiencies (IE) and collection efficiencies (CE)

Season	IE	RIE NH ₄ ⁺	RIE SO ₄ ²⁻	RIE Cl ⁻	CE
Pre-monsoon	2.92E-07	4	1.45	2.07	0.5
Monsoon	2.92E-07	4	1.45	2.07	0.5
Post-monsoon preflux period (11/10/18 - 03/11/18)	2.89E-07	4	1.45	2.07	0.5
Post-monsoon Diwali period (05/11/18 - 14/11/18)	3.14E-07	4	1.45	1.05	0.8
Post-monsoon post-Diwali (14/11/18 - 23/11/18)	3.14E-07	4	1.45	1.05	0.5

4. Line 124: I will try to mention how many data points were eventually considered and used to run PMF.

The number of data points within the organic matrix have been added: “The primary PMF analysis was conducted on the organic matrix (2.39673×10^7 data points)...”

5. Line 134-136: Not convinced about how you decide the CE=1 for the Diwali period. Why keep the gradient of 0.8 instead of 0.9, which you obtained from pre- and post monsoon campaigns?

The collection efficiency for the Diwali period has been changed to CE=0.8 to maintain the gradient of 0.9. This has resulted in a slight change in many averages and statistics, and all the relevant figures and tables have been updated accordingly.

6. Line 263-264: “The pollution rose suggests that most of the organic mass is from the east and southeast with high peaks ($> 140 \mu\text{g m}^{-3}$) originating from the west and northwest.” I will add “(Fig. 4(e))” at the end of the sentence. But I cannot draw the same conclusion as you do, I can only see that these high peaks were not originated from east south, northeast, and southwest, the width of the other direction at >140 looks quite similar to me. I will suggest to re-do the plot with more bins of concentration >140 to see if you can see some patterns. Otherwise, I will not make such a statement.

We have now provided a new version of the pollution rose figure for the organics (new Figure 3). There are now more meaningful breaks in the concentration bins and the sizing of the wind vectors have been changed to show the contribution of a particular wind direction to the mean concentration (rather than to the frequency of counts, as before). The statement has been altered to better reflect the data shown in the newly edited figure: “The pollution rose and polar graph for organic aerosol are highly spread. The pollution rose suggests that there is a slight increase in the organic mass when originating from the east and south east but this is closely followed by contributions from the west and northwest (Figure 3e).”

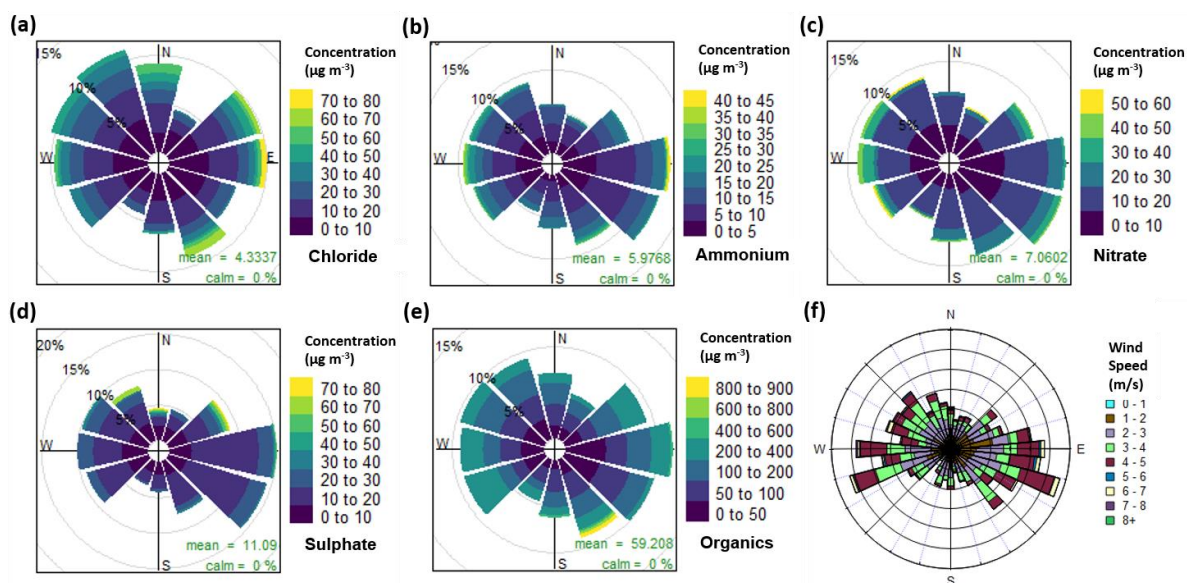


Figure 3 – Pollution roses for (a) chloride, (b) ammonium, (c) nitrate, (d) sulphate and (e) organic aerosol, along with (f) a wind rose plot for all measurement periods combined. The pollution roses show 30° wind vectors and their size is proportional to the percentage contribution to the mean concentration. The vectors are divided into concentration bins based on the colour scale in the legend.

7. Line 264: “Its polar graph also shows some extreme values existing from the southeast.” Add “(Fig. S8)” at the end of the sentence to direct readers. Please read through your manuscript again to guide the reader properly. We have added a reference to Figure S8: “Its polar graph also shows some extreme values existing from the south east (Figure S8).”

8. Figure 4: Why there are negative concentration in (b), (d), and (e), what happened to these data points? 1. These plots did not seem to have many fractions for the smallest concentration bin; 2. I am surprised that you still end up some noisy data points in such a polluted area like India, please explain in more details if you do believe these negative points are real.

We agree that these are incorrect and we found an error in the concentration scale which has been corrected. The pollution rose has also been altered to better reflect the direction of mass associated with wind direction. Previously the graphs were showing bin sizes based on frequency of counts in a particular wind direction but now they are sized to show the percentage contribution to the concentration mean. This creates a clearer picture of wind directional preferences (see new Figure 3).

9. Figure 6: Please explain what the uncalibrated PAH concentration is in the caption.

We have provided an explanation in the text when the PAH time series figure is first introduced: “The time series of PAH concentrations in Figure 4 are uncalibrated concentrations and further lab work (such as the work carried out in Herring et al. (2015)) is required to establish absolute concentrations which goes beyond the scope of this study.” Please note that Figure 6 is now Figure 4.

10. Line 299-300: “The PAH time series in Figure 6 shows similar quantities during the pre-monsoon and monsoon periods and the consistent low levels suggest common sources such as traffic, solid fuel burning or

cooking activities.” Similar to what? OrgNO? Is the second part of the sentence not finished yet? Or did you mean because the low level of PAH, suggested the sources of PAH are traffic, solid fuel burning, or cooking activities? If so, please add a citation at the end.

We apologise that the English was not quite correct here and we have altered the text to explain that PAHs are similar in quantity during the monsoon and pre-monsoon. Due to the fairly consistent level of PAHs, we suggest that the dominant sources during these periods are also consistent emitters such as traffic, solid fuel burning and cooking activities: “The data does, however, show the relative change in PAHs is small between the pre-monsoon and monsoon periods suggesting consistent sources are responsible such as traffic, solid fuel burning or cooking activities.”

11. Line 300-302 “There is however a large increase in PAHs during the post-monsoon period when the burning of the rice crop residue begins,” how do you know it started in the post-monsoon, please provide a citation here. A citation has been added to the statement (Kulkarni et al., 2020).

12. Figure 8: label the Diwali period.

The Diwali period has been labelled using a light blue shading in (see new Figure 6).

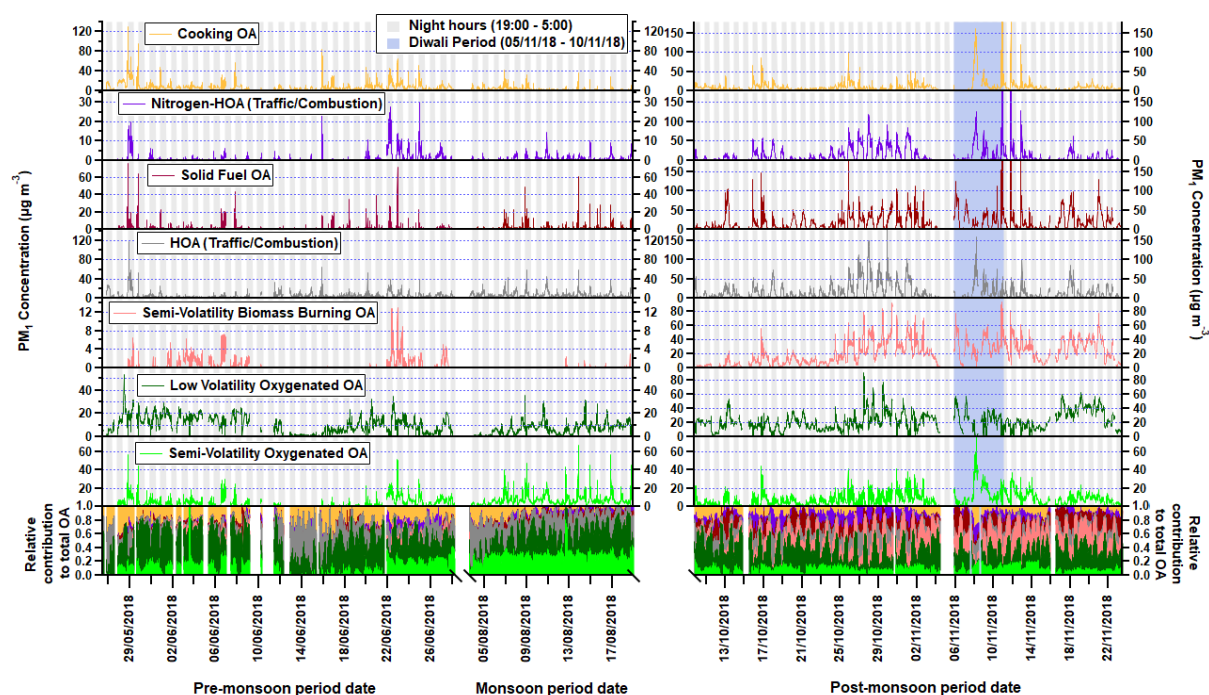


Figure 6 - Time series for each factor where the x-axis is broken to show each measurement period. Regions shaded in grey are night hours and the Diwali period is shaded in light blue. The time series of the normalised concentrations are also shown in the bottom section of the graph.

13. Line 345: “The PMF results from the AMS unit-mass resolution flux measurements (Ben Langford pers. commun.) show two traffic factors which peak at 9 a.m. and 11 a.m., which supports this.” There are some other source apportionment studies in India having HOA factor, I suggest not citing an unpublished manuscript.

We have updated the citation to a European Aerosol Conference abstract which summarised the results (Di Marco et al., 2019).

14. Figure 9: It is difficult to locate the “Lower panel” when it is a text not a “(b)” in the caption especially when the caption is so long. In addition, for the lower panel graph, I would change the y-axis to mass concentration, and keep the percentage of each factor inside the bar, it makes readers easier to compare the relative concentration of each factor for a different time period. Besides, I think you don’t gain extra information here by adding inorganic into this graph when you already have Figure 2.

The upper and lower panels on this figure now have additional (a) and (b) labels (see Figure 7). If the y-axis is altered to mass concentration in Figure 7(b), it becomes particularly hard to see changes between the monsoon and pre-monsoon as the post-monsoon is 3 times larger in mass and dominates the graph. The inorganics are added to show the total PM_{10} which makes it easier to view the fraction of total PM_{10} mass that is due to each factor. This gives factor mass more meaning and we are reluctant to remove the inorganics for this reason.

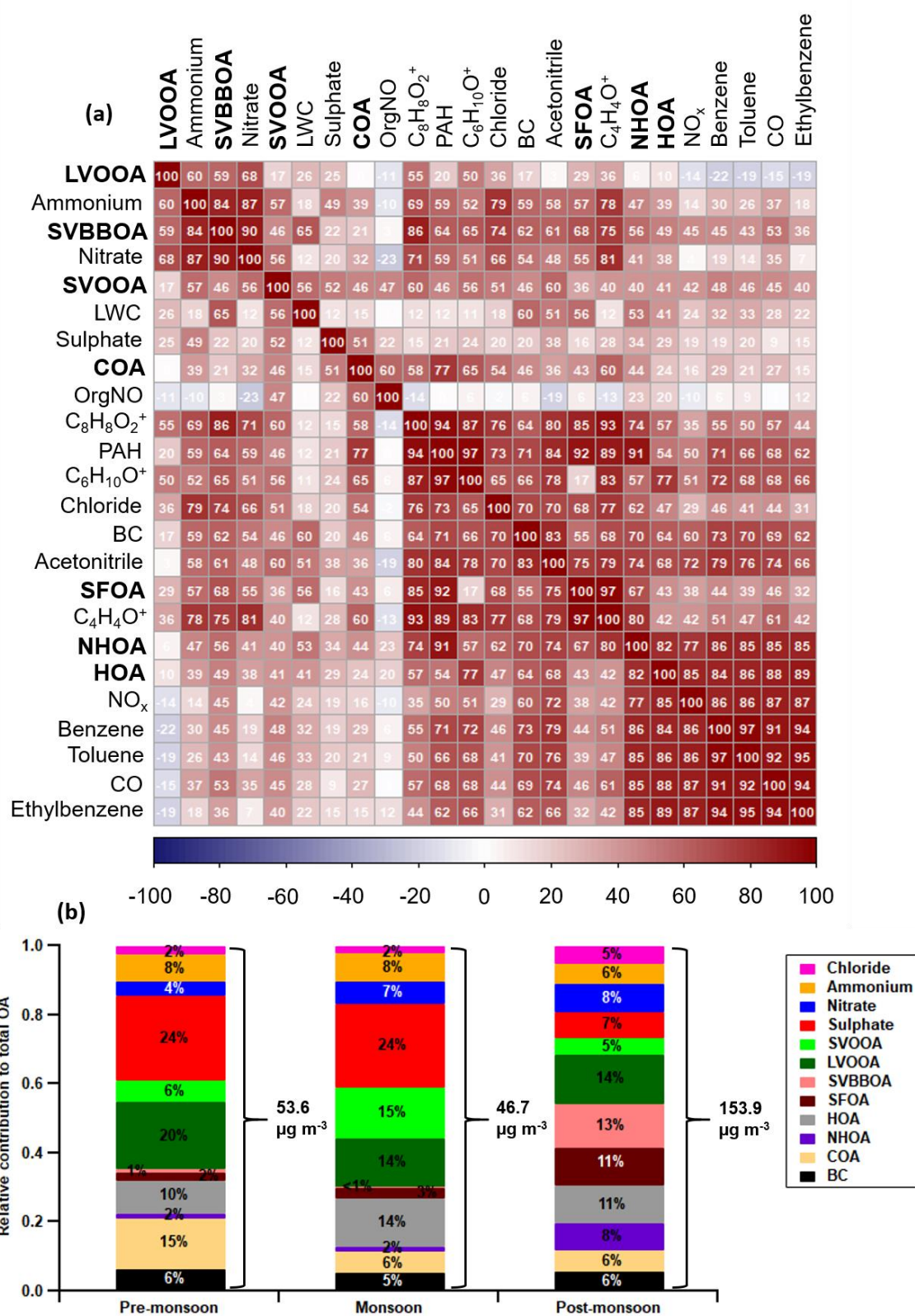


Figure 7 – Correlation matrix (a) between the AMS OA factors (bold), internal tracers and external tracers for the combined dataset (all seasons). The correlation coefficients are ordered using hierarchical cluster analysis. The relative contribution of OA factors, BC and inorganic species to total PM_{10} for each period (b) where the total average PM_{10} is shown using right curly brackets (see Table S5 for values and statistics). The three ions $[C_6H_{10}O]^+$, $[C_8H_8O_2]^+$ and $[C_4H_4O]^+$ are, respectively: an organic acid fragment used for COA determination, a fragment of dibenzodioxin called benzodioxan and, furan a fragment of dibenzofuran. The abbreviations of VOC species are: Ace = acetonitrile, Ben = benzene, Tol = Toluene and EBen = Ethylbenzene.

15. Line 364: *“This may suggest atmospheric processing is occurring, for example oxidation reactions.” Please explain what oxidation reactions occurred and why it caused the larger spread of the mass of NHOA in different wind directions. Or did you just simply imply the NHOA was rather fast oxidized that cannot transport in a long-range?*

We have added the following text to provide a clearer explanation: “A larger spread for NHOA when compared to HOA could suggest that atmospheric processing is occurring as both have similar traffic origins. NHOA could therefore be a result of aged or oxidised traffic emissions allowing it to be transported further distances and causing a mixture of wind directional preferences.”

16. Line 437-438: *“The polar graph also shows maximum concentrations coincide with a south-easterly direction. The timing of these peaks match with maxima seen in polar graphs for PAH, SFOA, and NHOA, which suggests a common source.” Please mention which figure it is since you have two polar graphs and two pollution rose figures.*

References to figures have been added within the text: “The polar graph also shows maximum concentrations coincide with a south-easterly direction (Figure S10h). The timing of these peaks match with maxima seen in polar graphs for PAH, SFOA and NHOA (Figure S10), which suggests a common source.”

17. Line 458: *“UnSubPAHs” is not defined in the previous text.*

This has now been defined within the text: “The PAH composition of COA is mainly unsubstituted PAHs (UnSubPAHs) and also contributes the largest...”

18. Line 836-837: *“One study indicates that this behaviour results in larger amounts of municipal waste being burnt in the morning compared to the evening (Nagpure et al., 2015).” One could add is that the temperature in India is not low, thus the evening peak is not so pronounced.*

We have added this observation to the following text: “One study indicates that this behaviour results in larger amounts of municipal waste being burnt in the morning compared to the evening (Nagpure et al., 2015). Additionally, the temperature in Delhi does not drop until later in the evening making it less likely that residents burn refuse to stay warm in the earlier parts of the evening.”

19. Line 925: *“PCDBs” and “PCDFs” did not mention previously.*

These have now been defined within the text: “We introduce novel AMS measured tracers which show the presence of polychlorinated dibenzo-furans (PCDF) and -dioxins (PCDD). This offers a new way to associate sources to plastic or Cl-rich fuel burning.”

Comments for SI:

1. Line numbers are missing

Line numbers have been added.

2. The second paragraph on P3: I understand previous studies used the terminology of “background concentration” to define the intercept of the multilinear regression, but I found it was difficult to read directly

Table S3 – A description of labels used to define a solution.

Label (Xf_ac_<120m/z_SX_CX)	Description
Xf, where X=1,2,3,n	The number of factors
ac	If present, this indicates it is resolved from the all-combined analysis case (all periods analysed in one PMF analysis). Otherwise, the solution is limited to a specific measurement period
<120mz	If present, this indicates the solution is limited to include ions up to m/z 120. Otherwise, ions up to m/z 385 are included
SX, where X=1,2,3,n	The SEED number
CX, where X=1,2,3,n	Indicates a specific combination of factors used for the <i>SFOA</i> variable in Eq. (S1) an <i>SFOA</i> factor time series alone is used for the <i>SFOA</i> variable C1 a combined time series of an <i>SFOA</i> and an <i>SVBBOA</i> factor C2 a combined time series of two <i>SFOA</i> factors (for solutions that produce two <i>SFOA</i> C3 factors)

4. Title on P7: “S2. Method for determining the Inorganic-Organic PMF solution” should be S3

The section heading has been changed.

5. Figure S8 on P13 and Figure S10 on P15: add the units for conc. and (a), (b), (c), (d), (e), etc. in the graph. Consider using a color code or anything else to differentiate the wind speed but not with closed circles. It is just not easy to read about the overlapped points, which are true for most of your data points.

Units and (a), (b), (c), etc. labels have been added to the figure (see Figure S8 and S10). When using a colour scale, the same issue occurs with overlapping of data points. The spread of the data is better presented in the revised pollution roses in the main text (new Figure 3 and 9). The polar plots are therefore only present to emphasise the maximum concentrations and their origin. This is highlighted in the SI text: “The polar plots offer additional information about the highest values which are seen more clearly in Figure S8 and show the direction of possible high source contributors.”

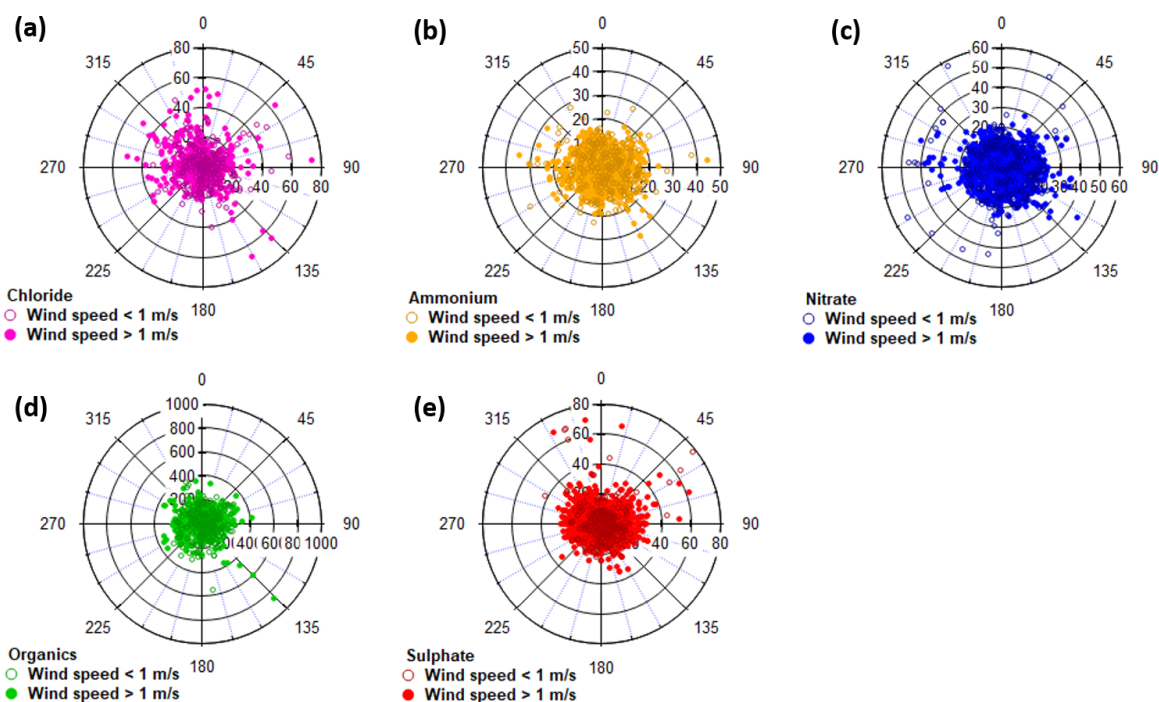


Figure S8 – Polar graphs showing the concentrations (in $\mu\text{g m}^{-3}$) by wind direction for chloride, ammonium, nitrate, sulphate and organics for all measurement periods combined. Each point represents a 5-minute measurement. Open symbols show concentrations for wind speeds $< 1 \text{ m s}^{-1}$ and closed symbols for wind speeds $> 1 \text{ m s}^{-1}$.

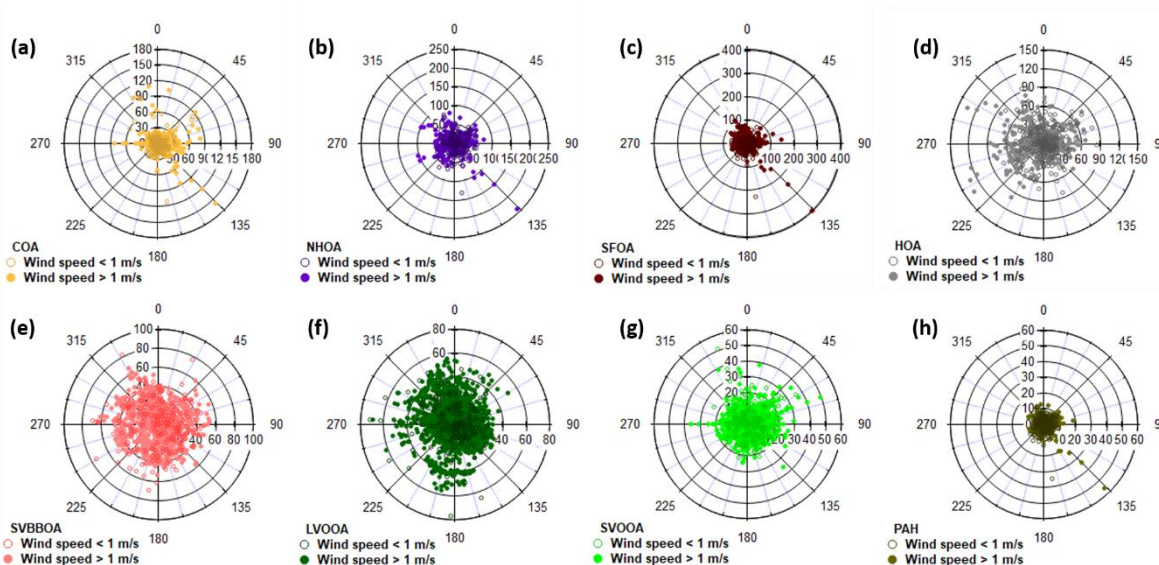


Figure S10 – Polar graphs showing the concentrations (in $\mu\text{g m}^{-3}$) by wind direction for the factor solutions: (a) COA, (b) NHOA, (c) SFOA, (d) HOA, (e) SVBBOA, (f) LVOOA and (g) SVOOA. (h) PAH polar graph shows uncalibrated concentrations (a.u.) by wind direction. Each point represents a 30-minute average measurement. Open symbols show concentrations for wind speeds $< 1 \text{ m s}^{-1}$ and closed symbols for wind speeds $> 1 \text{ m s}^{-1}$.

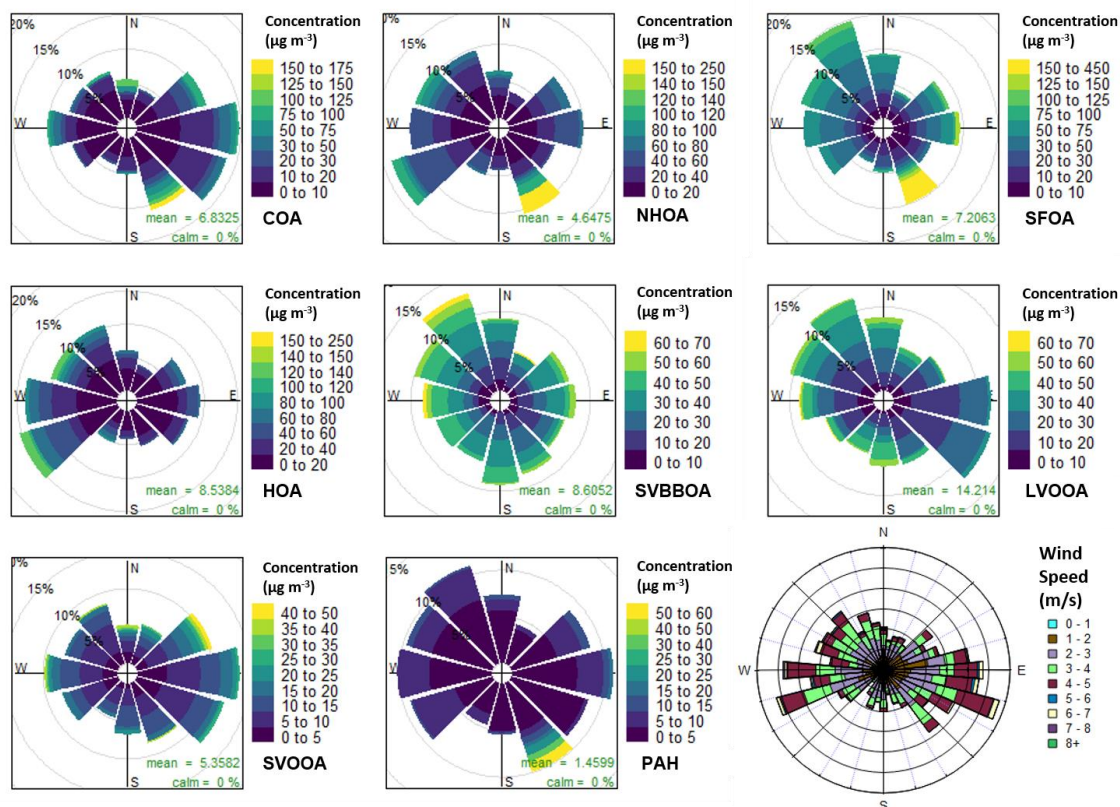


Figure 9 - Pollution roses for each factor and uncalibrated PAH concentrations along with a wind rose. The pollution roses show 30° wind vectors and their size is proportional to the percentage contribution to the mean concentration. The vectors are divided into concentration bins based on the colour scale in the legend.

6. The first paragraph on P12: “Error! Not a valid bookmark self-reference”, please revise.

Correction made.

7. Figure S7 on P19: Could you also label where the two cremation sites are if you are going to mention them in the text.

The cremation site has been labelled on Figure S16. Upon inspection of the second infant cremation site, we are unsure whether this site would truly contribute to PM measured at IGDUTW. This site is also particularly small in comparison to the very large cremation site nearby. We have therefore chosen to remove references to the infant cremation site.

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