Evolution of source attributed organic aerosols and gases in a megacity of central China

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Abstract. The secondary production of the oxygenated organic aerosol (OOA) impacts air quality, climate and human health. The importance of various sources in contributing to the OOA loading and associated different aging mechanisms remains to be elucidated. Here we present concurrent observation and factorization analysis on the mass spectra of organic aerosol (OA) by a high-resolution aerosol mass spectrometer and volatile organic compounds (VOCs) by a proton-transfer-reaction mass spectrometer in Wuhan, a megacity in central China during autumntime. The full mass spectra of organics with two principle anthropogenic sources were identified as the traffic and cooking sources, for their primary emission profiles in aerosol and gas phases, the evolutions, and their respective roles in producing OOA and secondary VOCs. Primary emissions in gas and aerosol phases both contributed to the production of OOA. The photooxidation of traffic sources from morning rush-hour caused 2.5-folds increase of OOA mass in a higher oxidation state (oxygen-to-carbon, O/C = 0.72), coproducing gas-phase carboxylic acids; while at night, cooking aerosols and VOCs (particularly acrolein and hexanal) importantly caused the nocturnal formation of oxygenated intermediate VOCs, increasing OOA mass by a factor of 1.7 (O/C = 0.42). The daytime and nighttime formation of secondary aerosols as contributed by different sources, was found to be modulated by solar radiation and air moisture respectively. The environmental policy should therefore consider the primary emissions and their respective ageing mechanisms influenced by meteorological conditions.

1 Introduction

The transformation between gas and aerosol phase of organic species produces secondary organic aerosols (SOA) (Kroll and Seinfeld, 2008; Hallquist et al., 2009; Slowik et al., 2010), which forms important global budget of aerosol loadings (Heald et al., 2005; Zhang et al., 2007), exerting climate (Poschl, 2005; Seinfeld et al., 2016) and environmental impacts (Von Schneidemesser et al., 2011; Huang et al., 2014). Given the large complexities of organic species, the yields (Goldstein and Galbally, 2007; Ortiz-Montalvo et al., 2014) and production rates (Sareen et al., 2013; Jokinen et al., 2015) of SOA from gas precursors are influenced by the diversities of source profiles (emission mass percentages among species) (Lin et al., 2012; Shrivastava et al., 2015) and environmental factors (such as radiation, air moisture and ambient temperature) (Clark et al., 2016; Li et al., 2021b; Wang et al., 2021). This raises challenges for source-oriented environmental policy making, until explicit understanding on the formation mechanism of SOA from different sources.

The application of detailed mass spectra of organic aerosols allows the online source attribution of organic aerosol (OA) (Canagaratna et al., 2007; Ng et al., 2011a), based on the factorization analysis on the mass spectra of OA which groups the covaried species from certain sources (Ulbrich et al., 2009). This factorization technique allows identification of primary sources and aged secondary sources at a receptor measurement site. The complex aerosol sources in urban environment have been commonly recognized as traffic sources mainly from vehicles on road (Zhu et al., 2021), biomass burning from open or closed combustion (Adler et al., 2011), coal combustion normally in cold season for heating (Xu et al., 2019) and more localized cooking sources (Allan et al., 2010; Zhang et al., 2020). The secondary sources are oxygenated OA which may be from oxidizing volatile organic compounds (VOCs) following condensation (Donahue et al., 2006), or heterogenous oxidation occurring on particle phase (Claeys et al., 2004). Among these sources, SOA has been found to be the main contributor of OA mass loading (41–69 %) in urban environment of East Asia (Sun et al., 2014; Hu et al., 2017), especially in warm season when primary emissions were low, along with high ambient temperature and more intensive chemical reactions (Hu et al., 2016). The formation of SOA from VOCs may experience a few reaction generations (Kroll and Seinfeld, 2008) and could interact with other sources of species during the process (Shrivastava et al., 2017), hereby complicating the goal of identifying the key precursors in contributing to the consequent SOA. In addition, some primary gases already have somehow low volatility and may not require a long reaction chain to become condensable, such as some primarily emitted intermediate volatility organic compounds (IVOCs) may substantially contribute to the SOA (Robinson et al., 2007; Huang et al., 2021). An understanding of source profiles of primary emissions in both gas and aerosol phases is therefore important to rule out the source-dependant production of SOA. The above necessitates the concurrent investigation on the compositions of gases and aerosols at a receptor site, along with their evolution and interaction, in order to elucidate the role of each source in contributing to SOA.

In this study, we performed online continuous measurements on the detailed mass spectra of organics concurrently on aerosol and gas phases, in a typical anthropogenically polluted region in central China, where such data had been rarely

64 reported. Factorization analysis is performed on the mass spectra of organics in both aerosol and gas phases, to investigate

65 the source-oriented gas-aerosol evolution and SOA formation in this region.

2 Materials and methods

2.1 Sampling site

68   The field experiment was performed in the campus of the China University of Geosciences (Wuhan) (114.40° E, 30.52°

69 N) during Oct.–Nov. 2019 (Hu et al., 2021). The site represents a typical region of mixed residential/traffic sources (Fig. S1).

70 Due to the preparation and hosting of the 7th CISM Military World Games during the experimental period, the government

71 implemented strict emission reduction measures, particularly for the industrial sources and heavy vehicle emissions in the

72 main roads. The more localized pollution sources, such as traffic emission from smaller sizes of vehicles and cooking

73 sources dominated the pollution in this study, besides those transported from surrounding regions during some heavy

74 pollution events (Zheng et al., 2019; Zheng et al., 2020). The HYSPLIT model (Draxler and Hess, 1997) with a 3-hourly 1°

75 $\times$ 1° wind field from the GDAS reanalysis products was used to obtain the 36-h backward trajectories initializing from the

76 location of the experiment site. The cluster analysis was performed to categorize the trajectories into three groups by

77 minimizing the differences in each group and maximizing the differences among groups (Moody and Galloway, 1988).

2.2 Instrumentation

2.2.1 Measurements of mass spectra of aerosols and gases

80   The mass concentration and chemical composition of NR-PM$_1$ were measured by a HR-ToF-AMS (Aerodyne Research

81 Inc., USA), including organics, nitrate (NO$_3^-$), sulphate (SO$_4^{2-}$), chloride (Chl$^-$), and ammonium (NH$_4^+$). The detailed can be

82 found elsewhere (Decarlo et al., 2006). Briefly, the aerosols are dried using a diffusion dryer before entering the AMS and

83 through a critical orifice into a narrow beam via an aerodynamic lens. The aerosol size is determined using the flight time of

84 particles to the thermal vaporization and ionization chamber. Then the aerosols are successively vaporized by a heated

85 surface ($\sim$600 ℃), ionized by electron ionization (EI, 70 eV), and detected by a mass spectrometer detector. During this field

86 observation, the HR-ToF-AMS was operated under V mode with high sensitivity. The composition-dependent collection

87 efficiencies were applied (Middlebrook et al., 2012), and the ionization efficiency was calibrated using 300 nm pure

88 ammonium nitrate (Jayne et al., 2000). Elemental analysis (EA) was also executed using the "Improved Ambient" method

89 (Canagaratna et al., 2015) to obtain the hydrogen-to-carbon ratio (H/C), oxygen-to-carbon ratio (O/C), and nitrogen-to-

90 carbon ratio (N/C).

91   The Proton-Transfer-Reactor Time-of-Flight Mass Spectrometer (PTR-ToF-MS 8000, Ionicon Analytik GmbH

92 Innsbruck, Austria) was deployed to quantify VOCs in this research. The operating and calibration of the PTR followed the

93 routine described previously (Cappellin et al., 2012; Bruns et al., 2016). Briefly, the PTR was operated with hydronium ions

94 (H₃O⁺) as the reagent and with a drift tube pressure of 2.2 mbar, voltage of 600 V and temperature of 60 °C. The ratio of the

95 electric field (E) and the density of the buffer gas (N) in the drift tube, which dictates the ion drift velocity in the drift tube, is

96 135 Td. MS transmission function was performed using a mixture of VOCs (formaldehyde, methanol, acetonitrile,

97 acetaldehyde, acetone, isoprene, methyl ethyl ketone, benzene, toluene, styrene, benzaldehyde, ethylbenzene, 1,3,5-

98 trimethylbenzene). Mass calibration was done using H_3O^+ (*m/z* 21.0226), $CH_3COCH_4^+$ (*m/z* 59.0490) and monoterpenes (*m/z*

99 137.1290). The shift in m/z is minor which ensures that the mass calibration was sufficient for all compounds. The

100 background measurements were performed using a dry zero air cylinder. The measurement error is described in Text S2. A

101 separate reaction rate constant is applied to convert the ion signal into concentration (Bruns et al., 2016), or the default

102 reaction rate constant 2×10^{-9} cm³ s⁻¹ can be applied to all other ions (Wang et al., 2020b). The vapor saturation concentration

103 (equilibrium vapor pressures) (C^*) of each VOC compound at 25 ºC is estimated using the parameterization based on

104 elemental ratio and molecular weight (Fig. S2) (Li et al., 2016). A logC^* (in μg m⁻³) lower than 6.5 is deemed to be

105 intermediate VOC (IVOC). According to Fig. S2, VOC species (with $m/z > 120$) are mainly identified to be IVOCs in this

106 study, thus the fraction of these larger molecular weight (MW) VOCs ($m/z > 120$) is used to evaluate the potential influence

107 of IVOCs.

108 **2.2.2 PMF analysis on the mass spectra of OA and VOCs**

109 Positive matrix factorization (PMF) (Paatero and Tapper, 1994) was performed on the high-resolution organic mass

110 spectral matrix of OA (Ulbrich et al., 2009; Decarlo et al., 2010). In this work, m/z > 120 and isotopic ions were excluded in

111 PMF analysis due to the limited mass resolution and low contributions to OA loading (~5 %). After a careful evaluation of

112 the mass spectral profiles and correlations with time series of tracers, diurnal variations, four OA factors from total OA were

113 identified with $f$peak = 0, including hydrocarbon-related OA (HOA), cooking OA (COA), low-oxidized oxygenated OA

114 (LO-OOA, OOA1), and more-oxidized oxygenated OA (MO-OOA, OOA2). The detail of PMF diagnostic was summarized

115 in Text S1 and Fig. S3.

116 The EPA PMF 5.0 model (Paatero and Tapper, 1994) was used for the source apportionment of VOC species. The 109

117 VOCs were used for the PMF analysis. The uncertainties from each sample were determined according to the method

118 detection limit (MDL) and the error fraction (%). The detail of PMF diagnostic was summarized in Text S2 and Fig. S4. Five

119 factors were ultimately selected, and the Q/Q_{exp} ratio was 0.96 on average (Fig. S4). The rotation ambiguity was explored by

120 varying the $f$peak values from -3 to +3, and the results with $f$peak = 0 were selected for the lowest dQ (robust), indicating the

121 stability of the PMF solution (Zhou et al., 2019). Most of the residuals are distributed normally, ranging from -3 and +3,

122 suggesting the model fits the data well.

123 **2.2.3 Other measurements**

124 The concentration of BC particles was measured by a single particle soot photometer (SP2, DMT Inc.). The operation

125 and data analysis procedures of the SP2 have been described elsewhere (Schwarz et al., 2008; Liu et al., 2010). The SP2

126   incandescence signal was calibrated for refractory BC (rBC) mass using the Aquadag black carbon particle standards

127   (Acheson Inc., USA) and corrected for ambient rBC with a factor of 0.75 (Laborde et al., 2012).

128   The size distribution and number concentration of aerosols with an electrical mobility diameter from 12 to 552 nm were

129   also measured by a scanning mobility particle sizer (SMPS, Model 3081, impactor 50 % cut off at 0.677 µm; CPC model

130   3775 at a flow rate 0.3 L min^{-1}). PM_1 mass concentration calculated based on the volume concentration measured by the

131   SMPS agreed well with that from the sum of compositions by the HR-ToF-AMS and SP2 (r = 0.71, slope = 0.90).

132   The gas-phase species including NO_x, O_3, and CO were measured in real time by a series of Wuhan Tianhong analyzers

133   (TH-2001H, TH-2003H, TH-2004H, respectively). These instruments were calibrated periodically with the corresponding

134   standard gas to ensure the accuracy of the observation data. In addition, the meteorological parameters including temperature

135   (T), relative humidity (RH), and wind speed and direction were recorded by an automatic weather station.

136   **3 Results and discussion**

137   **3.1 Chemical compositions of PM_1**

138   The time series of mass concentrations of BC, non-refractory-PM_1 (NR-PM_1) species (i.e., OA, SO_4^{2-}, NO_3^-, NH_4^+, and

139   Cl^-) and their relative contributions are summarized in Fig. 1. During this field observation, the mass concentrations of NR-

140   PM_1 were in the range of 2.5–44.8 µg m^{-3}, with an average of 12.7 ± 5.7 µg m^{-3}, which is close to those observed in the

141   North China Plain in autumn 2019 (15.1 µg m^{-3}) (Li et al., 2021a), and was much lower than that (41.3 ± 42.7 µg m^{-3})

142   observed in Beijing in autumn 2012 (Hu et al., 2017). The mean mass concentration of BC was 0.29 ± 0.17 µg m^{-3} with the

143   range of 0.1–1.0 µg m^{-3}. Among all species in NR-PM_1, OA contributed the major (49.2 %), indicating the dominant role of

144   OA in autumntime of PM_1 pollution in this region. Inorganic aerosol accounted for 50.8 % of NR-PM_1 in which sulfate was

145   the largest contributor (21.5 %), followed by nitrate (18.5 %), ammonium (10.7 %), and chloride (0.1 %), which has similar

146   relative contributions to those observed in field (Chen et al., 2021). The number concentration of all particles peaked at 95 ±

147   38 nm in with regular growth during every field day. Fig. 1 also presents the temporal evolution variation in meteorological

148   parameters. During the field observation period, the average temperature and relative humidity (RH) were 19.3 ± 3.6°C

149   (11.2–29.4 °C) and 75.0 % ± 17.5 % (25.0–99.0 %), respectively. The O_3 concentration with an average of 30.7 ± 13.0 ppb,

150   was highest 54.5 ppb at 15:00, likely due to the high temperature and enhanced photochemical processing (Zheng et al.,

151   2021).

152   Fig. 1f shows the spatial distribution of aerosol optical depth (AOD) and 36 h backward trajectories during the

153   campaign. Cluster-1 (C1) shows the circulated air mass with shortest transport distance for the past 36 h (36 % fraction); C2

154   is the northerly transported air mass (55 % fraction), over regions with higher AOD; C3 is the most rapid transport through

155 the northeast China and some coastal areas. It therefore separates the periods with more local air mass or influences from
156 regional transport. Fig. 1g shows the diurnal variations of solar radiation and RH, peaking in the day and night respectively.

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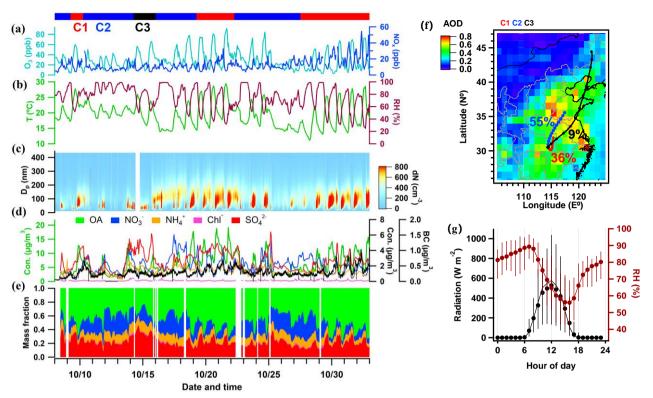
Figure 1: Time series of (a) mass concentrations of O₃ and NOₓ; (b) ambient temperature (T) and relative humidity (RH); (c) number size distribution measured by the SMPS; (d) mass concentrations of key aerosol species (the green left y-axis represents OA, the right y-axis represents other aerosol species); (e) mass fractions of chemical species in non-refractory PM₁; (f) spatial distribution of mean aerosol optical depth (AOD) during the experiment and the clustered 36 h backward trajectories; (g) diurnal profiles of direct solar radiation and RH.

3.2 Source attributed organic aerosol

164   The resolved OA factors by the PMF analysis on the AMS measurements are shown in Fig. 2. The first factor is
165 characterized by prominent hydrocarbon ion series of $C_nH_{2n-1}^+$ and $C_nH_{2n+1}^+$ (Fig. 2a), with a low O/C ratio (0.24), which is
166 generally considered to be related to the emissions of fossil fuel combustion and vehicle emissions (Huang et al., 2010;
167 Morgan et al., 2010; Ng et al., 2011b). The time series of the factor correlated well with NO_x (r = 0.71) and BC (r = 0.83)
168 (Fig. 2e and Table S1). The diurnal pattern of this factor showed peaks in the morning and afternoon rush-hour (Fig. 2i),
169 with a major increase from 5:30, reaching a peak value of 1.5 µg m⁻³ at 8:30. The concentration gradually decreased around
170 noontime due to boundary layer dilution until 15:00 and reached a minimum of 0.6 µg m⁻³. This spectrum also contained
171 some fragment markers for possible coal combustion OA (CCOA), i.e. $C_9H_7^+$ (*m/z* 115, r = 0.73) (Hu et al., 2013). This

172 factor was not distinctly resolved in this dataset maybe due to the urban nature of the site, where the traffic source may have

173 overwhelmed, due to the less significant coal combustion pollutants during the sampling period.

174   The second factor was characterized by m/z 55 ($C_4H_7^+$, $C_3H_3O^+$) and 57 ($C_4H_9^+$, $C_3H_5O^+$), accounting for 10 % and 3.5

175 % of the total spectrum, respectively (Fig. 2b), with the lowest O/C ratio among factors (0.16). This factor has a

176 $C_3H_3O^+/C_3H_5O^+$ of 3.0 and $C_4H_7^+/C_4H_9^+$ of 2.0 (1.0 is usually for HOA), indicating it as cooking source rather than HOA

177 (Mohr et al., 2012). The correlation coefficient of the COA factor and marker fragment $C_6H_{10}O^+$ was 0.91, which is also

178 similar to a previous study (Sun et al., 2011). The diurnal pattern of this factor showed a major peak during 18:00–20:00,

179 reaching up to 4.0 µg m^{-3} on average, in addition to a smaller peak at lunch time, which corroborated the diurnal cooking

180 activities. Previous studies reported COA accounted for 6.5–30 % of the total OA in urban (Rogge et al., 1991; Lanz et al.,

181 2008; Allan et al., 2010; Xu et al., 2014). Here the concentrations of COA in OA were in the range of 0.5–4.5 µg m^{-3} and

182 accounted for 23 % of OA mass fraction on average. The cooking emission in the studying region was likely from charcoal-

183 grilling activities, which was popular in the surrounding areas.

184   Besides the two primary OA, additional two oxygenated OA (OOA) factors are identified. According to the oxidation

185 state, OOA was separated into lower (OOA1) and more oxidized (OOA2) factors. OOA1 factor contained abundant oxygen-

186 containing fragments, accounting for more than 50 % of the total mass spectrum, with an O/C of 0.42. In particular, the

187 oxygenated fragment containing one oxygen accounted for 39 %. The $C_2H_3O^+$ ion (m/z 43) is an important component of less

188 oxidized SOA (Wang et al., 2020a), which was highly correlated with OOA1 (r = 0.94). OOA1 showed lower concentration

189 during daytime but higher concentration during nighttime (Fig. 2k). Such diurnal variation was similar to that of RH (r =

190 0.45), but opposite to that of solar radiation, which was similar to a previous report (Sun et al., 2014). OOA1 may thus be

191 associated with aqueous reactions when high RH. The co-occurrence of nighttime peak of OOA1 and COA suggested the

192 primary source of cooking emission may have considerably contributed to the production of OOA1.

193   The factor OOA2 had the highest O/C of 0.72 and contained 61 % oxygen-containing fragments (Fig. 2d), which is very

194 similar to the spectra of OOA factor resolved in other cities (0.60 and 67 % in Mexico City, 0.80 and 66 % in Pasadena)

195 (Aiken et al., 2009; Hayes et al., 2013). This factor was correlated strongly with the fragment of CO_2^+ (r = 0.79). Compared

196 to OOA1, this factor showed obvious diurnal variations with a major enhancement at around noontime (10:00–15:00), up to

197 2.9 µg m^{-3}, indicating the photochemical production of SOA during daytime. The variation of OOA2 correlated with odd

198 oxygen ($O_x = O_3 + NO_2$, r = 0.70), agreeing with previous observations that oxidized OA had strong correlation with O_x

199 (Wood et al., 2010; Hu et al., 2016). Considering the high RH during the experiment (> 60 %, Fig. 1g), OOA2 factor may

200 have also experienced aqueous chemistry and showed good correlation with sulfate (r = 0.82).
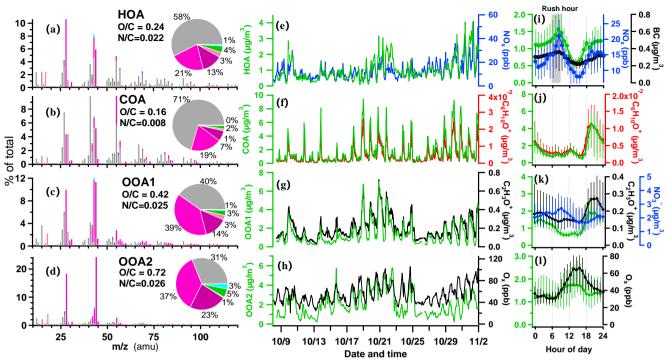
Figure 2: Results of source-attributed organic aerosols by the PMF analysis on the OA mass spectra measured by the HR-TOF-AMS. Mass spectra of PMF factors for (a) hydrocarbon-like OA (HOA); (b) cooking OA (COA); (c) oxygenated OA1 (OOA1); (d) oxygenated OA2 (OOA2). The pies show the relative contributions of the six ion categories to each factor. (e-h) Temporal variations of four OA factors (HOA, COA, OOA1 and OOA2) with each correlated species as NO_x, $C_6H_{10}O^+$, $C_2H_3O^+$ and O_x. (i-l) Diurnal profiles of NO_x, BC, $C_6H_{10}O^+$, $C_2H_3O^+$, NO_3^- and O_x. The lines and whiskers denote the median, the 25th and 75th percentiles at each hour, respectively.

3.3 Source attributed VOCs

Fig. 3 and Fig. S5 summarize the source attributed of VOCs and key indicators. Two primary and three secondary VOC factors were identified. Here 109 species of VOCs from the PTR-TOF-MS were used, which are mostly oxygenated or contain cyclic functional groups, in contrast with the GC-MS measurement which contains many aliphatic hydrocarbon compounds and usually only decades of species were used for the PMF analysis (Zheng et al., 2021). These may lead to some unsolved primary VOC sources for the current analysis using the PTR-measured VOCs, if the source contained crucial markers of aliphatic species, but this method may have great advantage in comprehensively resolving factors of secondary VOCs. This may explain the less primary but more secondary VOC factors compared to conventional PMF-resolved VOC sources (Cai et al., 2010).

The first factor was dominated by aromatic compounds, such as C_6H_6 (*m/z* 79.054) and C_7H_8 (*m/z* 93.070) in Fig. 3a. They were well established markers for vehicle emissions (Gkatzelis et al., 2021) and had a good correlation with this factor (r = 0.97 and 0.63, respectively Fig. S6). This factor showed peaks in the morning and afternoon rush-hour (Fig. 3k) and was

well correlated with HOA and NO_x (r = 0.63 and 0.53 Table S1). The concentration of this factor had a major increase during the early morning, reaching a peak value of 12.1 ± 1.3 ppb at 08:30, further corroborating the traffic source of this factor. The concentration decreased around noontime until 15:00 because of the dilution by well-developed boundary layer and in addition to its consumption through photochemical reaction due to intense solar radiation. The diurnal pattern was also high at night, although the peak was lower than early morning, the average concentration was higher by a factor of 2 than daytime. This suggests that traffic VOCs is more likely to participate in photochemical reaction, and other primary emissions may be precursors for nocturnal chemistry.

The second factor contained abundant aldehydes such as C_3H_4O (acrolein), $C_3H_6O_2$ (hydroxyacetone), $C_6H_{12}O$ (hexanal), and $C_7H_{14}O$ (heptanal) at m/z 57.069, 75.044, 101.096, and 115.112, respectively, as well as C_8H_{10} (C8-aromatics) and C_9H_{12} (C9-aromatics) at m/z 107.086 and 121.101 in VOC mass spectra (Fig. 3b), which are footprint VOCs identified from primary cooking emissions during the charbroiling and frying (Klein et al., 2016). This factor had similar time series (Fig. 3g) with COA and had high correlation (r = 0.67, Table S1). The concentration of this factor decreased during the daytime and increased after 18:00 with a peak value at 19:00 (17.2 ± 3.0 ppb). As shown in Fig. 3l, the diurnal concentration decreased strongly after emission and continued to decline throughout the night, suggesting that cooking VOCs may be major precursors and were consumed during night.

Besides the two primary VOCs, three secondary oxygenated VOCs (SecVOCs) factors are identified. These three factors are not well correlated with any primary factors attributed by the OA, are thus considered to be mainly contributed by secondary production. The daytime biogenic emission, e.g. isoprene, may also contribute to the SecVOC2 formation by interacting with OH in the presence of NO_x (Lin et al., 2013), producing methacrylic acid epoxide (MAE) and methacrolein (MACR) as intermediately involved in SOA formation (Gu et al., 2021). This factor was thus also contributed by this primary source. SecVOC2 contributed a large fraction of $C_2H_4O_2$, $C_3H_6O_3$ and $C_4H_6O_3$ (Fig. 3c), which were gas-phase carboxylic acids, the oxidation products from photochemical processes (Hartikainen et al., 2018; Li et al., 2021b). SecVOC2 had one clear peak at 13:00 (14.7 ± 2.8 ppb Fig. 3m). The time series of SecVOC2 correlated well with OOA2 (r = 0.67), following the variation of solar radiation. It had a rapid enhancement starting from 07:00 to 12:00 and declined continuously after 13:00. This indicated that many of these species can be formed rapidly during daytime and may have a short lifetime owing to the partitioning to the condensed phase and forming SOA.

SecVOC1 factor featured with some less-oxygenated VOCs e.g., $C_2H_2O_2$, $C_6H_{10}O_2$ and $C_{10}H_{14}O$ (Fig. 3d). This factor had a peak at 19:00 which was consistent with the primary cooking VOCs factor, but also increased throughout the night, peaking at midnight. This factor had a similar temporal trend with OOA1 (r = 0.76, Fig. 3i), which was less oxygenated than photochemistry dominated OOA2. Combining the features above, SecVOC1 tended to be contributed by some immediately reacted species from emissions in the late afternoon and early night. A particular factor (Fig. 3e) is significantly composed of large molecular weight (large-MW) oxidized VOCs, i.e. the mean contribution of ionic compounds with m/z > 120 was above 50 % in this factor (Fig. S5e), which was much higher than that in SecVOC2 (Fig. S5c). Fig. 3e shows its signature

253 compounds of $C_8H_{14}O$, $C_6H_{12}O_3$ and $C_8H_4O_3$, and some are nitrogen-containing VOCs, such as $C_6H_5NO_3$ and $C_8H_9NO_3$.

254 These VOCs with m/z > 120 tend to be intermediate-volatility organic compounds (IVOCs) as the estimated vapor saturation

255 concentration ($\log_{10}C^*$, 298 K) is less than 6.5 μg m^{-3} (Fig. S2). This factor is hereby termed as large-MW VOCs to indicate

256 the fraction of IVOCs, which only require few oxidation steps to become semi-volatile (Robinson et al., 2007). Fig. 3o

257 shows an increase of this factor at mid-night, later than the peak of SecVOC1, which may imply the ageing process in

258 producing these VOCs.

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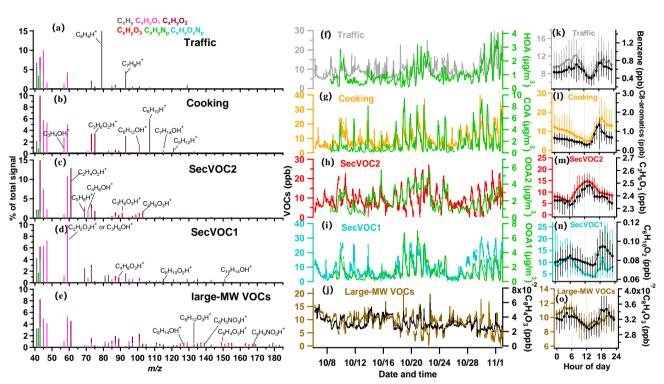

260
261 **Figure 3. Source attributed VOCs by the PMF analysis measured by the PTR-TOF-MS. (a-e) Mass spectra of the five factors**
262 **(traffic VOCs, cooking VOCs, secondary VOCs (SecVOC2, SecVOC1) and large molecular weight (MW) VOCs (large-MW**
263 **VOCs), with major fingerprint peaks labelled in the mass spectra. (f-j) Temporal variations of the five VOCs factors and their**
264 **respective correlated OA species. (k-o) Diurnal profiles of the VOC factors and their respective signature species. The lines and**
265 **whiskers denote the median, the 25th and 75th percentiles at each hour, respectively.**

266 **3.4 Oxidation process of organics in the day and night**

267   After source attribution of organics in aerosol and gas phase, we are able to identify the emission structure of primary

268 sources and the consequent evolution. Given the identified primary traffic and cooking sources were emitted in the day and

269 late afternoon respectively, this provides the potential opportunity to study the evolution of different primary sources in the

270 potentially contrasting ageing mechanisms in the day and night.

Fig. 4a-d shows the temporal evolution of the key factors in a week to reflect the day and night ageing. The O/C ratio in the day was higher than that at night by 0.2 (Fig. 4a), corresponding with the increase of highly oxygenated fragments of AMS f_{44} (CO_2^+) in the day (Fig. 4b). During night, the O/C ratio increased with the increase moderately oxygenated fragment f_{43} ($C_2H_3O^+$). The diurnal variation of atomic ratio O/C of OA (Fig. 4e) showed a slight decrease at morning rush-hour and sharp drop at 18:00 (by 0.2) due to the significant contribution of the traffic and cooking sources respectively, corresponding with the two steps of increase at each time. This variation of O/C clearly showed the daytime and nighttime ageing processes of OA for different primary sources. Notably, the O/C showed peak value of 0.54 at 15:00, implying the importance of photooxidation in producing highly oxygenated OA.

Considering the diurnal pattern of anthropogenic activities, the traffic emission at rush-hour is deemed to be the major source in contributing to the daytime production of OOA. The ratio OOA2/HOA is thus used to indicate the daytime oxidation of OOA. As Fig. 4d and 4f showed, OOA2/HOA had a clear peak during daytime, and increased after 8:00 and peaked at 14:00. After 15:00, the ratio gradually decreased to minimum at 20:00 and maintained to be low throughout the night. This clearly demonstrated the photooxidation in producing OOA2 from oxidizing HOA. Fig. 4c and 4g give a few examples of photooxidation in gas phase: the toluene showed a production of $C_3H_6O_3$ (hydroxypropionic acid) and $C_4H_6O_3$ (acetic anhydride) by a factor of 2 in 3 hours (Fig. S7c); the C_3H_4O (acrolein) produced the oxidized product $C_2H_2O_2$ (glyoxal) by a factor of 1.3 (Fig. 4h). All of these reacted species are from traffic VOCs and the corresponding products are from SecVOC2. The daytime biogenic emission, e.g. isoprene, may also contribute to the SecVOC2 formation by interacting with OH in the presence of NO_x (Lin et al., 2013), producing methacrylic acid epoxide (MAE) as intermediately involved in SOA formation, where a considerable nitrogen content was also found in the OOA2 factor (N/C = 0.026). Overall, the highly oxidized OOA2 (O/C = 0.72) is considered to be mainly contributed by the traffic source, via the oxidation of VOCs and partitioning to condensed phase, direct oxidation on HOA through heterogenous oxidation (Guo et al., 2020), or VOCs evaporated from HOA and further condensation after oxidation (Zhao et al., 2015). All factors may have contributed to the daytime production of OOA2.

The nocturnal oxidation is mainly contributed by the sources emitting from late afternoon throughout the midnight. Both traffic and cooking sources contributed to the emission since late afternoon, with cooking source as the predominant contributor in both aerosol (Fig. 2j) and gas (Fig. 3l) phases. The ratio between the nighttime OOA1 and cooking aerosol (OOA1/COA) is therefore used to indicate the nocturnal oxidation of SOA (Fig. 4d and j). There was a sudden increase of OOA1/COA during the daytime because COA was consumed rapidly in the afternoon after a small amount of emission at noon (Fig. 2j). The lowest OOA1/COA at 0.5 corresponded with the fresh cooking emission at 18:00, and kept increasing until peaking in the early morning at 6:00 up to 3, which was an increase by a factor of 6 compared to the minima (Fig. 4j). In addition to evidence for daytime reaction, Fig. 4h and Fig. S7d also gave evidence for certain reacted species during the ageing at nighttime. The first-generation oxidation products from acrolein (ACR $C_3H_4OH^+$, *m/z* 57.033) are glyoxal ($C_2H_2O_2$, *m/z* 59.036) and formaldehyde ($HCHOH^+$, *m/z* 31.018) according to the database in Master Chemical Mechanism

304   (MCM). This night oxidation is also evidenced by the formation of some nitrated organic compounds and ketone

305   compounds, such as $C_6H_5NO_3$ (nitrophenol, NP, Fig. 4l) and $C_6H_{10}O_2$ (hexanedione, Fig. 4k) produced from phenol (C_6H_6O)

306   and hexanal ($C_6H_{12}O$), respectively. Notably, an important fraction of large molecular weight-VOCs (which are mostly

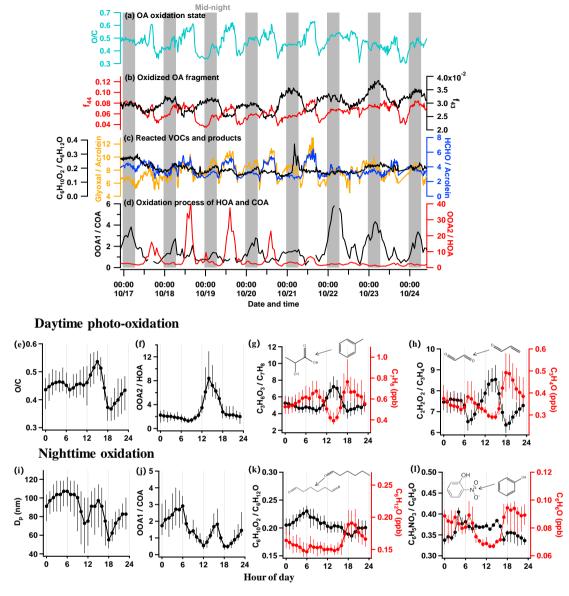
307   intermediate VOCs, Fig. S2) peaked at 3:00–4:00 (Fig. 3o), consistent with the variation of OOA1/COA during night. Some

308   of these nitrated and oxygenated IVOCs may have been further oxidized and partitioned to aerosol phase, contributing to the

309   OOA1. Given the larger molecular usually has a lower O/C ratio (Hatch et al., 2017) (because of a higher content of carbon),

310   this may explain the lower O/C observed for nighttime formed OOA1 (O/C = 0.42), than OOA2 produced by daytime

311   photooxidation (O/C = 0.72). Notably, nighttime SOA had a high N/C (0.025), implying the $NO_3\cdot$-initiated from cooking

312   emissions oxidation, which was different from the organic nitrate formation mechanism during the daytime.

313
314 **Figure 4. Time series showing the aging of aerosols and gases, with grey vertical bars denoting nighttime (00:00−06:00), (a) the**
315 **oxygen to carbon ratio (O/C); (b) the fragment fraction at *m/z* 44 (f_{44}), *m/z* 43 (f_{43}); (c) concentration ratio of $C_6H_{10}O_2$ to $C_6H_{12}O$**
316 **(hexanedione to hexanal), glyoxal to acrolein, and formaldehyde to acrolein; (d) concentration ratio of OOA1 to COA and OOA2**
317 **to HOA. Diurnal variations of key species showing the daytime photooxidation and nighttime oxidation, (e) ratio of O/C; (f) ratio**
318 **of OOA2 to HOA; (g) concentration of C_7H_8 and ratio of $C_3H_6O_3$ to C_7H_8 (hydroxypropionic acid to toluene); (h) concentration of**
319 **C_3H_4O and ratio of $C_2H_2O_2$ to C_3H_4O (glyoxal to acrolein); (i) particle diameter (D_p); (j) ratio of OOA1 to COA; (k) concentration**
320 **of $C_6H_{12}O$ and ratio of $C_6H_{10}O_2$ to $C_6H_{12}O$ (hexanedione to hexanal); (l) concentration of C_6H_6O and ratio of $C_6H_5NO_3$ to C_6H_6O**
321 **(nitrophenol to phenol).**

322

323   Fig. 5 summarizes the key indicators for the contrasting daytime and nighttime oxidation process. The concentrations

324 are normalized by ΔCO,   where ΔCO is the total CO concentration subtracted by the background concentration (1th

325 percentile of the dataset), to indicate the variation of species regardless of the boundary layer evolution, wind speed, wind

326 direction and temperature (Gouw and Jimenez, 2009). The odd oxygen O_x (O_3 + NO_2) has been widely used to generally

327 indicate the activity of daytime photochemistry (Hu et al., 2017), and the enhanced moisture is the main driving factor for

328 nighttime chemistry. The O_x concentration and RH are therefore used as references with which the variations of species were

329 correlated in the day and night, respectively. As Fig. 5a showed, the traffic primary emissions in both gas (traffic VOCs) and

330 aerosol phase (HOA) declined with increased O_x by 60 % and 40 % respectively, suggesting their roles as precursors in the

331 daytime reaction. The produced species are oxygenated SecVOC2 and OOA2, showing enhancement with O_x, peaking at

332 midday-afternoon. This process was rapid as the SOA production by a factor of 2.5 and the oxygenated VOCs production by

333 a factor of 1.7 within 6 hours. The traffic VOCs are widely observed to contribute to SOA production, with aromatic

334 compounds serving as key precursors (Fang et al., 2021). The semi-volatile nature of HOA means it could be evaporated to

335 gas phase and further oxidized to recondense as SOA (Robinson et al., 2007). The decrease rate of HOA with increased

336 photochemical age was also found in urban environment (Zhu et al., 2021), generally consistent with the reaction rate in this

337 study. Here we linked the declining rate as a function of photochemical activities for both reacted aerosol and gas phases for

338 traffic sources. The gases evaporated from aerosol phase (especially under higher temperature when increased saturation

339 pressure for semi-volatile or intermediate volatile species) and primary VOCs may be simultaneously involved in the

340 photooxidation, further contributing to the SOA formation.

341   For nocturnal oxidation shown in Fig. 5b, the reacted species are cooking VOCs and COA (decrease by 35 % and 77 %,

342 respectively), producing SecVOC1 and large-MW oxygenated VOCs, with an increase of night SOA formation by a factor

343 of 1.7. The nocturnal processes may have largely involved aqueous reactions, because the variations of reacted or produced

344 species were highly correlated with RH (r > 0.95, Fig. 5b). The large-MW VOCs (mostly IVOCs) increased by 50 % and

345 reached maxima when highest RH. This suggests the moisture may have been involved in converting some primary VOCs to

346 IVOCs, which further contribute to the SOA production during nighttime. Previous studies also found the oxidation of

347 IVOCs from cooking sources can be an important source of SOA (Zhang et al., 2020). The evidence is given here that

348 organic aerosols and gases from cooking emission had been reacted and contributed to SOA. An approximate production rate

349 of 0.2 µg m^{-3} h^{-1} of OOA from cooking source can be obtained by considering the ageing time of ~10 h (from COA peak

350 18:00 to OOA1 peak 4:00), which was generally consistent with previous laboratory work using gas precursors from cooking

351 sources (0.07–0.5 µg m^{-3} h^{-1}) (Liu et al., 2017). Notably, daytime SOA had a higher oxidation state, implying the importance

352 of photooxidation in producing highly oxidized OA. This may be because of the high temperature at daytime and a species

353 may require a lower volatility (hereby more oxygenated) to be in condensed phase than at night.

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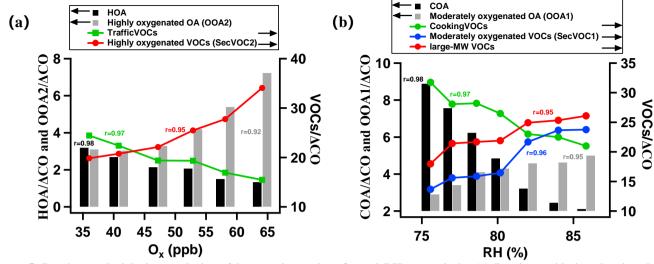
Figure 5. Daytime and nighttime evolution of key species against O_x and RH respectively. (a) Daytime oxidation showing the primary emissions of traffic source in gas and aerosol phases including HOA, traffic VOCs; and secondary products including SecVOC2 and OOA2, with concentrations all normalized by ΔCO. (b) Nighttime oxidation showing the primary emissions of cooking source in gas and aerosol phases including COA and cooking VOCs; and secondary products including SecVOC1, large-MW VOC and OOA1, with concentrations all normalized by ΔCO.

4 Conclusion

In this study, organic gases and aerosols were concurrently characterized through online mass spectrometers at a megacity in central China. Through the factorization analysis on the organic mass spectra, two principal sources-the traffic and cooking sources were identified for both aerosol and gas phases, hereby the reacted and produced species between phases were interlinked. We observed clear evidence of daytime and nighttime oxidation of source-attributed OA and VOCs. Daytime photooxidation caused 60 % decrease of primary aerosol and 40 % of primary VOCs reduction for traffic sources, producing oxygenated SecVOCs by a factor of 1.7 and OOA by a factor of 2.5, in a 6 hours photochemical ageing. Nocturnal ageing caused a reduction of primary OA (by 77 %) and primary VOCs (by 35 %) from cooking sources, producing oxygenated VOCs and OOA by a factor of 1.4 and 1.7, respectively. In particular, larger molecular IVOCs produced (by a factor of 1.7) at night may importantly contribute to the OOA. This implies primary species in aerosol and gas phases both contribute to the production of OOA. A higher oxidation state of OOA from daytime photooxidation was found than nighttime, suggesting different compositions of produced OOA modulated by solar radiation and moisture, respectively. Vehicle and cooking emissions are the major contributors of organic aerosols in urban areas, especially in megacities. These results provide direct observations about the reaction rate for primary precursors and production rate for secondary aerosols, as influenced by primary sources and meteorological conditions. The environmental policy making should therefore consider the respective primary sources and ageing mechanisms for local and regional atmospheric

377 environmental problems. The particular regulation should be placed to avoid the formation of daytime highly-oxidized
378 species when high solar radiation, which may contribute to the reactive oxygen species and exert adverse health impacts
379 (Tao et al., 2003; Verma et al., 2009).

380 **Data availability**

381   The data in this study are available from the corresponding author upon request.

382 **Author contribution**

383   DL, SK, and SL led and designed the study. SL, YW, HZ, YC, SZ and DH set up and conducted the experiment. SL,
384 DL, YW, KH, XJ and SD contributed to the data analysis. QL, DZ, JS provided technical support and assistance. SL and DL
385 wrote the manuscript. All authors read and approved the final manuscript.

386 **Competing interests**

387   The authors declare no competing interests.

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