



This discussion paper is/has been under review for the journal Atmospheric Chemistry and Physics (ACP). Please refer to the corresponding final paper in ACP if available.

Burning of olive tree branches

E. Kostenidou et al.

Burning of olive tree branches: a major organic aerosol source in the Mediterranean

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Received: 23 February 2013 – Accepted: 1 March 2013 – Published: 19 March 2013

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Published by Copernicus Publications on behalf of the European Geosciences Union.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Abstract

Aerosol produced during the burning of olive tree branches was characterized with both direct source-sampling (using a mobile smog chamber) and with ambient measurements during the burning season. The fresh particles were composed of 80% organic matter, 8–10% black carbon (BC), 5% potassium, 3–4% sulfate, 2–3% nitrate and 0.8% chloride. Almost half of the fresh olive tree branches burning organic aerosol (otBB-OA) consisted of alkane groups. Their mode diameter was close to 70 nm. The oxygen to carbon (O : C) ratio of the fresh otBB-OA was 0.29 ± 0.04 . The mass fraction of levoglucosan in PM_1 was 0.034–0.043, relatively low in comparison with most fuel types. This may lead to an underestimation of the otBB-OA contribution if levoglucosan is being used as a wood burning tracer. Chemical aging was observed during smog chamber experiments, as f_{44} and O : C ratio increased, due to reactions with OH radicals and O_3 . The otBB-OA AMS mass spectrum differs from the other published biomass burning spectra, with a main difference at m/z 60, used as levoglucosan tracer. In addition to particles, volatile organic compounds (VOCs) such as methanol, acetonitrile, acrolein, benzene, toluene and xylenes are also emitted. Positive matrix factorization (PMF) was applied to the ambient organic aerosol data and 3 factors could be identified: OOA (oxygenated organic aerosol, 55%), HOA (hydrocarbon-like organic aerosol, 11.3%) and otBB-OA 33.7%. The fresh chamber otBB-OA AMS spectrum is close to the PMF otBB-OA spectrum and resembles the ambient mass spectrum during olive tree branches burning periods. We estimated an otBB-OA emission factor of $3.5 \pm 0.2 \text{ g kg}^{-1}$. Assuming that half of the olive tree branches pruned is burned in Greece 2280 ± 140 tons of otBB-OA are emitted every year. This activity is one of the most important fine aerosol sources during the winter months in the Mediterranean countries.

ACPD

13, 7223–7266, 2013

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



1 Introduction

95 % of the global biomass burning is anthropogenic in an effort to clear land, convert forests to agricultural and pastoral lands, heat residences and to dispose of agricultural waste (Crutzen and Andreae, 1990; Schüle, 1990; Andreae and Merlet, 2001).

5 Biomass burning can contribute up to 90 % of fine particulate matter in the burning season in the Amazon (Yamasoe et al., 2000). During the winter, biomass combustion is one of the major sources of organic aerosol in large parts of Europe (Puxbaum et al., 2007). The release height of the emissions is low and the mixing during the winter months is often weak, leading to deterioration of the local air quality and visibility
10 and increase of the risk of adverse effects on human health especially in populated areas (Lighty et al., 2000; Boman et al., 2003; Naeher et al., 2007). Biomass burning particles also affect earth's albedo by light-scattering (Crutzen and Andreae, 1990; Andreae, 1995) and can act as cloud condensation nuclei (CCN), modifying the cloud droplet size distribution and therefore the reflectivity of clouds (Rogers et al., 1991; Roberts et al., 2002).

15 Numerous laboratory-based studies have been conducted for the physical and chemical characterization of biomass combustion aerosol (e.g. Shafizadeh, 1984; Abas et al., 1995; Simoneit et al., 1999; Hennigan et al., 2010). The fresh biomass burning particles are in the submicron size range and consist of approximately 80 % organic compounds, 5–10 % black carbon (BC) and 10–15 % inorganic salts (potassium, sulfate, chloride and nitrate) (Reid et al., 2005). The most abundant individual compound identified by GC-MS analysis is levoglucosan (1,6-anhydro- β -D-glucopyranose, $C_6H_{10}O_5$, a product of cellulose thermal decomposition (Shafizadeh et al., 1973, 1979, 1984). Schneider et al. (2006) reported that AMS mass spectra of beech,
20 oak, musasa, spruce and savanna grass burning are dominated by hydrocarbon fragment ions $C_nH_{2n-1}^+$ ($m/z = 27, 41, 55, 69, \dots$), $C_nH_{2n+1}^+$ ($m/z = 29, 43, 57, \dots$) and $C_nH_{2n-3}^+$ ($m/z = 67, 81, 95, \dots$). These mass spectra contained characteristic levoglucosan markers at m/z 60 (0.7–4.4 % of the total organic signal) and at m/z 73 (0.8–2.2 % of the

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



total organic signal). Weimer et al. (2008) found that during the smoldering phase the biomass burning particles are dominated by oxygenated species and they proposed the mass fragments 29, 60 and 73 as levoglucosan signatures. A number of organic vapours are emitted during biomass burning, together with the particles. For example during laboratory biomass burning Christian et al. (2003) quantified the emissions of gases such as acetaldehyde, phenol, acetol, glycolaldehyde, acetone, furan, acetonitrile, toluene and benzene.

Several ground and aircraft campaigns have focused on biomass burning (e.g. Fang et al., 1999; Hudson et al., 2004; Crouse et al., 2009; Yokelson et al., 2009; Pratt et al., 2011). Anhydrosugars such as levoglucosan, manosan, galactosan, arabinosan and xylosan have been identified by GC-MS in aqueous extracts of aerosol samples from pasture and rainforest sites in Rondônia, Brazil (Graham et al., 2002). Alfarrá et al. (2007) suggested the mass fragments 60, 73 and 137 as AMS marker fragments for wood burning aerosols based on measurements in Switzerland. Elsasser et al. (2012) indicated a high correlation between the wood combustion organic aerosol factor using positive matrix factorization (PMF) and the levoglucosan measured with one-hour resolution PM₁ filters by GC-MS during winter measurements in Augsburg, Germany. During airborne measurements of the savanna fires in Africa (Yokelson et al., 2003) numerous gas phase compounds were identified, among them CO, methane, methanol, formaldehyde, formic acid, acetic acid, etc.

The emission factors, EFs, of biomass burning compounds have been estimated in various studies. Sinha et al. (2003) found that the EF of the organic particulate carbon during savanna fires in southern Africa was $2.3 \pm 1.2 \text{ gC kg}^{-1}$ and that the major organic emitted gas-phase compound was acetic acid with $\text{EF} = 2.4 \pm 0.9 \text{ g kg}^{-1}$. In the same study the EF of the number of particles from 0.1 to 3 μm was $2.0 \pm 1.5 \times 10^{14} \text{ kg}^{-1}$ of fuel burned. The EFs of toluene and benzene have been reported to be in the 0.13–0.79 and 0.18–0.24 g kg^{-1} range correspondingly (Andeae and Merlet 2001; Sinha et al., 2003; Christian et al., 2003). CO is also one of the most abundant compounds

emitted during biomass burning fires. The reported EFs values are in the range 65–137 g kg⁻¹ (Andea and Merlet, 2001; Christian et al., 2003; Yokelson et al., 2009).

The majority of the previous studies have focused on either wild fires or on combustion of wood that is used for residential heating during the winter. However, there is little information in the literature about burning of agricultural waste and more specifically olive tree branches burning. Burning of olive tree branches is a common agricultural waste management technique after the pruning of olive trees during the months November to February. Almost 1 billion (90 %) of the olive trees in our planet are located around the Mediterranean. Forty years ago Darley et al. (1974) estimated that the EF of olive tree wood burning of fine PM is around 5 g kg⁻¹. Using Greece as an example (132 million olive trees) and assuming that around 10 kg of branches are pruned from one olive tree, 6600 tons of fine PM will be emitted in a 3–4 month period in Greece. Comparing this with the 150 tons of fine PM per year produced from the 5 000 000 passenger cars in Greece, assuming 30 g of fine PM per year (EPA, 2005), the emissions of olive tree branches burning in Greece and the Mediterranean Area can be a major source of fine aerosols during the late fall and winter.

The present work will focus on the characterization of both particles and vapours emitted during olive tree branches burning and the quantification of the corresponding emissions. We will combine source sampling and characterization (including chemical aging of the emissions in a smog chamber) with field measurements during the burning season.

2 Experimental procedure

2.1 Source sampling – chamber experiments

Four olive tree branches burning experiments (Table 1) were carried out in the Institute of Chemical Engineering Sciences (ICE-HT) campus. Approximately 15 kg of branches from the nearby fields were used for each experiment. The fire was initiated using

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



a butane torch and a pile of approximately 5 kg of branches. The rest of the branches were gradually added to the fire mimicking the procedure used by the farmers. In experiment 4 a piece of plastic was burned together with the branches, something that often happens during the field fires. A 10 m³ Teflon chamber was filled with smoke for 15–20 min using insulated 0.5 in copper tubing and a pump (model MB 602, Metal Bellow). After partial filling the chamber was moved back to the laboratory where its contents were diluted by a factor of 10 to 25 with particle and pollutant free air until its volume reached 10 m³. A suite of instrumentation was then used for the characterization of the particle and gas phase for 10–20 h.

2.2 Instrumentation

2.2.1 HR-ToF-AMS

The size-resolved chemical composition of the aerosol was monitored using a HR-ToF-AMS from Aerodyne Research Inc. (DeCarlo et al., 2006). The HR-AMS measures only the non-refractory PM₁ species. The vaporizer temperature was set at 600 °C and the tungsten filament for electron ionisation was run at an accelerating voltage of 70 eV. The HR-AMS run alternatively between V-mode (single stage reflectron) and W-mode (double stage reflectron) with 3 min of measuring time for each mode. In this paper the V-mode data are presented.

2.2.2 PTR-MS

A Proton Transfer Reaction Mass Spectrometer (PTR-MS, Ionicon Analytik) was used for the characterization of the volatile organic compounds (VOCs). The PTR-MS sampled continuously in the selected mass scan mode (23–42 masses were selected) with a total sample time of 6–10 s. A Teflon filter (Whatman PTFE 0.2 μm) was installed before the inlet tube in order to minimize the particles entering the instrument. The inlet tube was at 60 °C and the same temperature was used for the reaction chamber. The

drift tube pressure was 2.2 mbar and the voltage applied was 600 V. For the calibrations a standard mixture of 17 VOCs (Ionicon Analytik) was used.

2.2.3 SMPS

A Scanning Mobility Particle Sizer, (SMPS, classifier model 3080, DMA model 3081, CPC model 3775 or 3787, TSI) operated at a sheath flow rate of 5 L min⁻¹ and a sample flow rate of 1 L min⁻¹. The SMPS measured the number size distribution in the 10–500 nm range.

2.2.4 MAAP

A Multiple-Angle Absorption Photometer (MAAP, Thermo Scientific Inc.) was used during 2 chamber experiments for the BC measurement. The MAAP measures the amount of light (wavelength 637 nm) that is transmitted and scattered back from a particle laden glass fibre filter (Petzold and Schönlinner, 2004).

2.2.5 Gas monitors

A suite of gas monitors was used for the measurement of the NO_x, O₃, CO and CO₂ concentrations (Teledyne, models T360, 400E, 300E and T360) respectively. A series of gas standards (Air Liquid) was used for the calibration.

2.2.6 GC-MS

For some chamber experiments teflon filters (Whatman PTFE, 0.2 μm, 47 mm), quartz (Pall Tissuquartz, 47 mm) and high volume quartz filters (Whatman QMA, 101.6 mm) were collected. For the 47 mm filters a sampler equipped with a PM_{2.5} inlet was used (MetOne SuperSASS) and for the 101.6 mm filters a high volume sampler (220 L min⁻¹, Tisch Environmental) was used. The corresponding samples were analyzed by gas chromatography mass spectroscopy (GC-MS, Shimadzu QP2010 Ultra)

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



and the levoglucosan concentration was quantified. The extraction procedure and analysis conditions are similar to that of Simpson et al. (2004). Details for the analysis are included in the Supplement (SI).

2.2.7 FTIR

5 During 2 experiments filters for Fourier Transform Infrared Spectroscopy (FTIR) functional group analysis were collected directly from the fire and from the chamber after dilution (Maria et al., 2003; Gilardoni et al., 2007). The samples were analyzed using an FTIR Spectrometer (Bruker Tensor 27 spectrometer with DTGS detector). FTIR spectra were collected by averaging 128 absorbance scans at wavenumbers from 400 to
10 4000 cm^{-1} with a resolution of 2 cm^{-1} . The filters were scanned prior to use, and these spectra were subtracted from the spectra obtained after sampling. More information about the method followed can be found in Maria et al. (2002).

2.3 Ambient measurements

15 Sampling of ambient aerosols was conducted in the area of the ICE-HT Institute, which is located 8 km east of Patras and is surrounded by olive tree fields. The measurements took place from November 2011 to February 2012 (olive tree branches burning period) with sampling over a total of 20 days. The instrumentation used has been described in Sect. 2.2 and included the HR-AMS, the PTR-MS, the SMPS, the MAAP and the NO_x , O_3 , CO and CO_2 monitors.

2.4 Data analysis

20 HR-AMS data analysis was performed using the standard HR-AMS data analysis software SQUIRREL v1.51C and PIKA v1.10C (Sueper, 2011) with Igor Pro 6.22A (Wave-metrics). For the HR-AMS organic mass spectra we used the fragmentation table of Aiken et al. (2008) with the following modifications at m/z 's 28 and 18. The m/z 28 may
25 include contributions from both the gas and particle phase. The PToF spectra of m/z 28

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

I◀

▶I

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



showed that there was a large air beam signal (from N_2^+). However, there was no clear organic particulate signal. The signal of the particulate phase was very noisy, so the organic aerosol contribution at m/z 28 was set to zero. The m/z 18 may be influenced by water and organic compounds. The organic aerosol (OA) at m/z 18 was set equal to the OA signal at m/z 44, because the size distribution of the m/z 18 was similar with the size distribution of m/z 44, though noisier. Using this modification the particle bound water signal was positive.

During the chamber experiments the species measured by the HR-AMS were, organics, sulfate, nitrate, chloride and potassium. However, the HR-AMS can only detect a fraction of the potassium, and thus only the relative values are reliable. Using the BC concentrations measured by the MAAP we estimated the BC mass distributions assuming a size-independent OA/BC ratio. Applying the algorithm of Kostenidou et al. (2007), we estimated a collection efficiency (CE) of 0.6 ± 0.05 for the chamber experiments. This is consistent with other biomass burning CE estimations (Huffman et al., 2005; Capes et al., 2008). This value was also used for the ambient measurements.

PMF (Paatero and Tapper, 1994; Lanz et al., 2007) is a bilinear unmixing model used to describe the measurements as a linear combination of factors. The model solves the problem: $\mathbf{X} = \mathbf{G}\mathbf{F} + \mathbf{E}$ where \mathbf{X} is a $a \times b$ matrix of the organic mass spectra for a time periods and with b m/z 's, \mathbf{F} is a $p \times b$ matrix p profile factors (which are constant mass spectra), \mathbf{G} is a $a \times p$ matrix with the corresponding contribution of each factor and \mathbf{E} is an $a \times b$ matrix of the residuals. The PMF minimizes the sum of the squared and uncertainty-scaled residuals and then the \mathbf{G} and \mathbf{F} are fit (Paatero and Tapper, 1994). In this work we applied PMF to the ambient measured AMS organic mass spectra. As inputs we used data from 20 days from November 2011 to February 2012, when olive tree branches fires were observed. PMF was applied for the m/z 's 12–200 to both unit mass resolution (UMR) and high resolution (HR) organic mass spectra, following the approach of Ulbrich et al. (2009).

3 Olive branches burning aerosol characterization

3.1 Chemical composition

The fresh particles consisted of 80 % organic matter, 8–10 % BC, 5 % potassium, 3–4 % sulfate, 2–3 % nitrate, 0.8 % chloride and practically zero ammonium. For experiment 4, where plastic was burned together with the olive tree branches the CE was lower (0.45 ± 0.04) and the particle composition was different: 52 % organic matter, 5 % BC, 8 % potassium, 3 % sulfate, 2 % nitrate, 21 % chloride and 9 % ammonium.

The O : C ratio of fresh otBB-OA was estimated using the first hour of each experiment. It was on average 0.29 ± 0.04 which is close to the middle of the literature range 0.14–0.42 (Schneider et al., 2006; Weimer et al., 2008). Organonitrate compounds were also detected but with a low contribution of 5 ± 1 % of the organic mass. The presence of the organonitrates did not have a major impact (1–2 % difference) on the O : C ratio.

The composition estimated based on FTIR analysis (experiment 2), indicates that the otBB-OA was composed of approximately 49 % alkane groups, 28 % organic hydroxyl groups, 10 % carboxylic acids, 10 % primary amine groups and 3 % carbonyl groups (by organic mass) (Fig. S1). The normalized FTIR spectrum is shown in Fig. 1. Comparison with FTIR spectra from other BB-OA sources indicates that the otBB-OA spectrum has substantially higher organic hydroxyl group concentration and substantially lower carbonyl group. The higher hydroxyl group in the fresh otBB-OA is consistent with a contribution from vegetative detritus (consisting of cellulose) (Liu et al., 2012). The lower carbonyl groups in the fresh BBOA are also expected, as atmospheric processing of biomass burning aerosol has been shown to increase ketone groups and decrease hydroxyl groups (Hawkins and Russell, 2010). In addition, the differences in the alkane group region are expected given that the biomass precursors in Mexico and Texas likely do not include olive trees. As a result, the fresh otBB-OA has a weak correlation (Fig. 1) with BBOA measured in Mexico City ($R^2 = 0.34$), at Paseo de Cortez ($R^2 = 0.29$), and at Houston ($R^2 = 0.21$) (Liu et al., 2009; Russell et al., 2009).

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Teflon PM_{2.5} and Quartz total PM filters were analyzed by GC-MS for the measurement of levoglucosan. For the Teflon filters the ratio of levoglucosan to PM_{2.5} mass was 0.027–0.035 and for the Quartz filters the ratio of levoglucosan to total PM mass was 0.017–0.025. Calculating the OC from the AMS the levoglucosan to PM₁ OC mass ratio was between 0.034 and 0.043. For hardwood and softwood combustion the reported levoglucosan/OC ratios are between 0.01 and 0.334 (Fine et al., 2001; Fine et al., 2004). Puxbaum et al. (2007) reported a levoglucosan/OC ratio of 0.08 for beech wood and 0.21 for spruce wood. The levoglucosan/OC ratio for oak was around 0.06 (Sullivan et al., 2008). The ratio measured for the olive tree braches is close to the lower limit of the above reported values. This implies that otBB-OA contains less levoglucosan than BBOA from burning of other types of wood or vegetation.

The average normalized organic mass spectra of the 4 olive tree branches burns at the beginning of the sampling are shown in Fig. 2. The mass spectrum was characterized mostly by m/z 's 27, 29, 39, 41, 43, 44, 55, 57, 67, 69 and 91. High resolution mass spectra analysis showed the co-existence of hydrocarbon fragment ions and oxygenated species for the homologous chains C_nH_{2n-1}⁺ and C_nH_{2n-3}O⁺ at m/z = 27, 41, 55, 69 and the homologous chains C_nH_{2n+1}⁺ and C_nH_{2n-1}O⁺ at m/z = 29, 43, 57. The signal of the m/z 44 (CO₂⁺), which is a marker for OOA (Zhang et al., 2005), was pronounced in the mass spectrum. The sampled otBB-OA included particles produced during both the flaming and smoldering phases. Weimer et al. (2008) found that the flaming phase was dominated by hydrocarbon fragment ions such as C_nH_{2n-1}⁺ (m/z = 27, 41, 55, 69, etc.) and C_nH_{2n+1}⁺ (m/z = 29, 43, 57, 71, etc.) while the smoldering stage had much higher CO₂⁺ contribution. In experiment 2 m/z 44 was quite lower than m/z 43 probably because during this experiment the contribution of the flaming phase was higher. There was also signal above m/z 101 with characteristic peaks at m/z 105, 107, 115, etc. (Fig. 2b). The organic signal in the m/z range 101–300 accounted for 21 % of the total. The levoglucosan markers m/z 60 (C₂H₄O₂⁺) and m/z 73 (C₃H₅O₂⁺) (Schneider et al., 2006) were low (0.65 % and 0.76 % correspondingly) in all

4 experiments. The contribution of $C_2H_4O_2^+$ and $C_3H_5O_2^+$ to the m/z 's 60 and 73 was around 90 % and 60 % respectively of the corresponding organic signal at these m/z 's.

The fresh otBB-OA AMS mass spectra in the four experiments were quite similar to each other despite the rather different conditions. The angles θ between the vectors corresponding to the AMS spectra varied from 5 to 13.2 degrees (Table S1). In experiment 4, plastic was burned in addition to the olive tree branches, something that often happens in open olive tree branches fires in the field. Though the inorganic composition was different from the other experiments e.g. chloride was 21 % and also ammonium was detected as well, the organic composition was very similar to experiments 1, 2 and 3.

Figure 3 depicts the angle θ between the organic mass spectra of the otBB-OA and 27 other biomass burning organic aerosol (BBOA) spectra in the AMS mass spectral data base (<http://cires.colorado.edu/jimenez-group/AMSSd/>). The otBB-OA spectrum was quite different from most of the BBOA mass spectra. Two of the closest spectra were those of Puerto Rican mixed wood and ceanothus with an angle of 19–21 degrees. One of the notable differences between the otBB-OA and other BBOA spectra is the contribution of m/z 60, the levoglucosan tracer. Figure 4 shows the contribution of m/z 60 in the BBOA mass spectra, with the otBB-OA spectrum having one of the lowest f_{60} fractions. This is consistent with the levoglucosan measurements by GC-MS analysis described above. The fraction f_{39} , mostly $C_3H_3^+$, of otBB-OA is also lower than in other BBOA spectra (in those that m/z 39 has been corrected through the fragmentation table instead of being zero). On the other hand the f_{37} (C_3H^+), f_{63} (mostly $C_5H_3^+$), f_{67} ($C_3H_7^+$) and f_{91} (mostly $C_7H_7^+$) in the otBB-OA spectrum are higher than in most other BBOA (not shown).

3.2 Chemical aging of otBB-OA

During the olive tree branches combustion gases such as O_3 , NO , NO_2 and CO , were also produced. The initial O_3 levels in the dark chamber after dilution varied from 45 to 230 ppb (Table 1). There was considerable NO_x (30–200 ppb) and CO (3 to 10 ppm)

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



also present (not shown). OH levels (Table 1) were estimated using the decay of m/z 93 (toluene concentration) in the PTR-MS assuming a second order reaction (Barnett et al., 2012). The OH concentrations calculated, were $0.4\text{--}1.0 \times 10^6$ molecules cm^{-3} . Thus, there were two oxidants in the chamber (O_3 and OH) in all experiments.

Figure 5 illustrates the time series of f_{44} and f_{43} during experiment 2. Time zero denotes the time when dilution ended. The f_{44} increased with time, while the f_{43} decreased and this behavior was observed in all experiments. For experiment 2 the O : C at $t = 0$ was approximately 0.22 but after 12 h it increased to 0.4 (not shown). These are indications of chemical aging that is taking place due to reactions of the otBB-OA with OH radicals and ozone. Figure 6 depicts the angle θ between the initial organic mass spectrum (at $t = 0$) with the organic mass spectra through out the experiment for all experiments. The mass spectra changed with the time. For example in experiment 2 the angle between the initial and the final mass spectrum is around 45 degrees. This is the biggest difference probably due to the higher initial ozone and OH concentration in experiment 2 and the thus the higher degree of aging. Figure S2 compares the HR mass spectra at $t = 0$ and $t = 11.5$ h for experiment 2, where the f_{44} (CO_2^+) has increased while the hydrocarbon part of the m/z 's: 29, 39, 41, 43, 55, 57, 67, 69, 71 etc. has decreased.

During this experiment FTIR filters were collected directly from the fire and from the chamber during the first hour of the experiment. FTIR analysis revealed that the carboxylic acid groups during the first hour in the chamber increased to 12.2 % compared to the carboxylic acid groups directly emitted from the fire (10.3 %) as shown in Figure S1.

3.3 Volatile organic compounds

A number of mass to charge ratios were monitored by the PTR-MS. Due to the limitations present in the identification of the specific VOCs exclusively through PTR-MS (DeGouw et al., 2003), only certain species are reported (Table 2), which have been found to dominate the specific m/z during previous biomass burning studies (Christian et al.,

Burning of olive tree branches

E. Kostenidou et al.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

2003; DeGouw et al., 2003; Karl et al., 2007). The major compounds detected from olive tree branches burning were: methanol (m/z 33), acetonitrile (m/z 42), acrolein (m/z 57), benzene (m/z 79), toluene (m/z 93) and xylenes (m/z 107). However, high signal was observed at additional m/z 's, for which either multiple species are reported in the literature at analogous concentrations or there was no standard compound available for calibration. Methanol, benzene, toluene and formaldehyde have been identified in fires in southern Africa, in Yucatan and in laboratory biomass burning (Sinha et al., 2003; Yokelson et al., 2003 and 2009; Christian et al., 2003). In experiment 2, where the sample was dominated by the flaming phase the VOC concentrations were 5 to 20 times higher than in experiment 1, which was a mixture of flaming and smoldering phase. In experiment 2 the branches burned were quite wet, and this resulted in a prolonged ignition phase. Thus the prolonged evaporation of water during this phase could have enhanced the VOC emission from the fuel.

4 Ambient measurements during the burning season

4.1 Chemical composition and mass spectra

Figure 7 shows the PM_{10} non-refractory composition measured by the HR-AMS from 9–14 December, 2011 (5 out of the 20 sampling days). For all the ambient data set we applied an average CE of 0.6 (based on chamber experiments). During the burning season (20 days of sampling) the aerosol as measured by the HR-AMS was composed of organics 58.8 %, sulfate 30.2 %, ammonium 7.5 %, nitrate 3.2 % and chloride 0.7 %. On average the organic mass concentration for the same period was $5.73 \mu\text{g m}^{-3}$, the sulfate was $2.96 \mu\text{g m}^{-3}$ and the ammonium was $0.73 \mu\text{g m}^{-3}$. The average O : C ratio was around 0.59, which is typical for winter in southern Europe. For example the O : C ratio during the winter of 2009 at Finokalia, Greece, was 0.54 (Hildebrandt et al., 2011). The average ozone concentration was 36.8 ppb and the average fraction of fragment m/z 44 was 0.12.

Burning of olive tree branches

E. Kostenidou et al.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



The vast majority of the high organic PM periods were due to olive tree branches burning in the fields around the ICE-HT Institute, reaching a maximum $\text{PM}_{1\text{-NR}}$ concentration of around $40 \mu\text{g m}^{-3}$. Chloride for the fire periods correlated with potassium ($R^2 = 0.47$) indicating the emission of potassium chloride. The organic mass spectrum during the fire events was very similar to the fresh average spectrum measured in the chamber experiments. Figure 8 shows a comparison of the average mass spectrum from the chamber to the average mass spectrum on 11 December 2012 09:00–12:00 a.m. ($\theta = 13.6$ degrees) where a fire was observed within a distance of 1 km. The ambient mass spectrum was characterized by the m/z 's 27, 29, 39, 41, 43, 44, 55, 57, 69 and 91.

4.2 Positive matrix factorization

Multiple PMF solutions were examined and evaluated for both the unit mass resolution (UMR) and the HR organic mass spectra. A 3-factor PMF solution was found to describe best the ambient OA AMS composition variation. The factors corresponded to otBB-OA, HOA and OOA. The correlation coefficient between the UMR spectra and the HR spectra (for the same PMF factor) are 0.92, 0.99 and 0.88 for OOA, HOA and otBB-OA respectively. Since the HR mass spectra provide more information than the UMR the HR solution is presented here. More details about PMF analysis are provided in Sect. 6 of the SI. Figure 9 shows the spectra of the 3 factors, OOA, HOA and otBB-OA and Fig. 10 shows the 3 factor time series for the 20 days. On average the organic matter consisted of 55 % OOA, 11.3 % HOA and 33.7 % otBB-OA. This implies that the burning of olive tree branches contributes significantly to the average OA during the burning season. The average otBB-OA organic mass concentration was approximately $1.9 \mu\text{g m}^{-3}$ for the 20 sampling days, but during the fire events it reached concentrations up to $27 \mu\text{g m}^{-3}$. During the same period in the center of Patras, approximately 8 km east, the organic mass concentration was around $20 \mu\text{g m}^{-3}$ based on filter measurements (Pikridas et al., 2013). This suggests that the impact of these burns extends to tens of kilometers and probably to even larger scales.

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



The O : C ratio of the otBB-OA mass spectrum (PMF) was 0.27 which is consistent with the average chamber O : C ratio of the fresh emissions (0.29 ± 0.04). This value is a little higher than other BBOA sources (e.g. Lanz et al., 2007; Mohr et al., 2012). The HOA mass spectrum is characterized by low O : C ratio (0.05) which is in the range of previous studies. For example the HOA factor of Mohr et al. (2012) the O : C ratio was 0.03, while in the HOA mass spectrum reported by Ulbrich et al. (2009) it was 0.18. The OOA spectrum had the highest O : C ratio (0.65), which suggests a high degree of oxidation.

The OOA factor time series correlates very well with sulfate ($R^2 = 0.56$), ammonium ($R^2 = 0.73$) and the CO_2^+ fragment (m/z 44) ($R^2 = 0.87$) (Table S2), which is typical for OOA (e.g. Hildebrandt et al., 2011; Elsasser et al., 2012). The sulfate correlation here is somehow lower than the ammonium probably due to its emission during fire periods as potassium sulfate. Nitrate concentrations were very low during the non fire periods. The hydrocarbon parts of the m/z 43 (C_3H_7^+), 55 (C_4H_7^+), and 57 (C_4H_9^+), that have been suggested as primary organic emission fingerprints, correlate well with the HOA factor ($R^2 = 0.88$, $R^2 = 0.79$ and $R^2 = 0.97$ correspondingly). Additionally the non-oxygenated fragment of the m/z 69 (C_5H_9^+), shows a strong correlation with the HOA concentrations ($R^2 = 0.92$).

Potassium and chloride correlated well with otBB-OA ($R^2 = 0.34$ and $R^2 = 0.53$) further confirming that potassium chloride is emitted during olive tree branches fires (Pratt et al., 2011). Nitrate also has a good correlation with otBB-OA ($R^2 = 0.61$), as potassium nitrate is emitted as well (Pratt et al., 2011). Although the contribution of the levoglucosan markers $\text{C}_2\text{H}_4\text{O}_2^+$ at m/z 60 and $\text{C}_3\text{H}_5\text{O}_2^+$ at m/z 73 is low, we find that these ions have a good correlation with the otBB-OA factor ($R^2 = 0.87$ for both). The oxygenated part of the m/z 55 ($\text{C}_3\text{H}_3\text{O}^+$), 57 ($\text{C}_3\text{H}_5\text{O}^+$) and 69 ($\text{C}_4\text{H}_5\text{O}^+$), also correlates well with the otBB-OA factor ($R^2 = 0.73$, $R^2 = 0.83$ and $R^2 = 0.66$ correspondingly). Further, otBB-OA correlates with the hydrocarbon part of the ions 27 (C_2H_3^+), 29 (C_2H_5^+) and 41 (C_3H_5^+) ($R^2 = 0.74$, $R^2 = 0.70$ and $R^2 = 0.69$ correspondingly). Fragments belonging to the homologous chain $\text{C}_n\text{H}_{n-2}^+$ also correlate with the otBB-OA (Table S2),

with the ions 89 ($C_7H_5^+$) and 115 ($C_9H_7^+$) being closer ($R^2 = 0.81$ and $R^2 = 0.88$). The $C_nH_{n-2}^+$ fragments have a weak correlation with the HOA factor ($R^2 = 0.07$ – 0.17) and they could be used as otBB-OA tracers.

In Table 3 we compare the 3 profiles with selected PMF factors found in the literature (Zhang et al., 2005; Lanz et al., 2007, 2008; Ulbrich et al., 2009; Hildebrandt et al., 2011; Mohr et al., 2012; Crippa et al., 2012). For example the angle θ between our OOA and the OOA estimated by Lanz et al. (2008) for winter at Zurich is 20.8 while the θ between our HOA and the HOA found by Mohr et al. (2012) in Barcelona is 10.8. The θ between the otBB-OA extracted from this work and the OB-OA factor from FAME-09 (Hildebrandt et al., 2011) is quite high. This difference might be due to the very low concentrations at Finokalia during the winter campaign of FAME-09 that may result in noisier mass spectra or/and due to additional biomass burning at Finokalia (e.g. grass).

The PMF otBB-OA spectrum and the chamber otBB-OA spectrum are very alike resulting in a θ of 10.6. This suggests that the ambient otBB-OA that was sampled had undergone relatively little processing and that our source samples are representative of the otBB-OA emitted to the atmosphere.

Figure 11 illustrates the ambient and the chamber OA data in the Ng triangle (Ng et al., 2010). Our ambient winter ICE-HT OA falls within the triangle. The chamber otBB-OA and the otBB-OA derived by the PMF analysis fall near the bottom left side of the triangle.

4.3 Size distributions

Figure 12 shows the size distributions measured during experiment 1. The vacuum aerodynamic diameter mode for the AMS was around 240 nm (Fig. 12b). The ambient mass distribution during a fire event, for example on 11 December 2012 09:00–12:00 a.m., had a peak at 300 nm (not shown). The mass mode diameter is higher in the ambient case because there are also aged, larger particles present together with those from the biomass burning. For the chamber experiments we used the algorithm of

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Kostenidou et al. (2007) and we estimated an otBB-OA density of $1.25 \pm 0.16 \text{ g cm}^{-3}$ for experiments 1, 2 and 3. For experiment 4, where plastic was burned along with the olive tree branches, the estimated otBB-OA density was much lower $0.81 \pm 0.06 \text{ g cm}^{-3}$.

Figure 13 shows the number distributions for the ambient data set during 25–30 November 2011, as measured by the SMPS, together with the otBB-OA time series PMF factor. The ambient size distributions during the olive tree branches fires had a number mode mobility diameter, around 60–80 nm, smaller than the mode diameter found in the chamber experiments which was around 120 nm (Fig. 12a). This could not be entirely due to the pump losses during the chamber filling. Using 2 SMPS systems we calculated the pump losses measuring ambient and laboratory generated (e.g. using an atomizer) particles (Fig. S10). However, reconstructing the chamber size distributions the mode diameter drops on average only 10 %, i.e. 110 nm, (Fig. 12a) which is still higher than the mode diameter when olive fires occur in the fields.

One explanation could be the initial high number concentration in the chamber ($250\,000\text{--}500\,000 \text{ particles cm}^{-3}$) which leads to rapid coagulation. The ambient number concentrations were less than $60\,000 \text{ particles cm}^{-3}$. To investigate this we used the TOMAS (Two-Moment Sectional Algorithm, Adams and Seinfeld, 2002) model using as initial condition the ambient otBB-OA size distributions scaled to $500\,000 \text{ particles cm}^{-3}$ and simulated only coagulation. The simulation showed that the otBB-OA particles coagulated fast and within 1–2 h reached a mode diameter of 115 nm (Fig. S11). The duration between the filling of the chamber and the end of the dilution was approximately 1.5 h. Therefore the growth of the particles in our chamber can be explained by coagulation of the fresh particles. The ambient distribution (mode at 70 nm, $\sigma = 0.4$) is the appropriate one for the fresh emissions by this source.

It is worth mentioning that there was no nucleation observed in any of the 4 experiments, however without adding any oxidation agents or using UV lamps. Hennigan et al. (2012) observed new particle formation within 30–60 min after initiating photo-oxidation (using UV lamps) on biomass burning emissions.

Burning of olive tree branches

E. Kostenidou et al.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

4.4 Particle and vapor emissions

The emission factor EF_i of the species i emitted during the open olive tree branches fires was estimated by:

$$EF_i = \left[\frac{(C_i)_p - (C_i)_b}{(CO_2)_p - (CO_2)_b} \right] EF_{CO_2} \quad (1)$$

5 where $(C_i)_p$ and $(CO_2)_p$ are the species i and CO_2 concentrations in the plume, $(C_i)_b$ and $(CO_2)_b$ are the corresponding concentrations in the background, i.e. the average level before and after the fire plume and EF_{CO_2} is the emission factor of CO_2 , that is the mass of CO_2 emitted per mass of olive tree branches burned. The EF_{CO_2} is $1600 \pm 100 \text{ g kg}^{-1}$ for various types of woods (e.g. Kaufmann et al., 1992; Andea
10 and Merlet, 2001; Christian et al., 2003; Yokelson et al., 2003, 2009). The olive tree branches burning plumes were indentified using the otBB-OA PMF factor.

On average the EF for the otBB-OA was estimated to be $3.45 \pm 0.2 \text{ g kg}^{-1}$ (Table 2), which is lower than the EF calculated from Darley et al. (1974). According to this estimate 4250–4820 tons of fine organic aerosol will be produced during the winter months
15 in Greece, which is quite high compared for example to the annual emissions of the passenger cars (150 tons per year). Assuming a low EF_{CO_2} of 1000 g kg^{-1} the otBB-OA EF would be $2.14 \pm 0.21 \text{ g kg}^{-1}$ and the emitted otBB-OA will be around 2820 tons, which is still a significant amount.

The nitrate EF was in the range $0.25\text{--}0.28 \text{ g kg}^{-1}$, which is consistent with the
20 measurements of Sihna et al. (2003) and Yokelson et al. (2009) for savanna fires in Africa and tropical forests in Yucatan. The sulfate EF was in the range $0.33\text{--}0.37 \text{ g kg}^{-1}$, higher than other sulfate EF's (Table 2), while the BC EF was 0.15 g kg^{-1} , lower than published values. For particles in the range $10\text{--}500 \text{ nm}$ the EF was equal to $3.5 \pm 0.35 \times 10^{15} \text{ particles kg}^{-1}$. This is higher than the EF reported by Sihna et al. (2003) $2.0 \pm 1.5 \times 10^{14} \text{ particles kg}^{-1}$, probably because their EF was estimated for larger particles ($0.1\text{--}3 \mu\text{m}$). The EF of CO is lower than in other studies,
25

40 ± 2.5 g kg⁻¹, while the benzene and toluene EF's are quite close to the EF's calculated by Sihna et al. (2003). Methanol and acetonitrile EF's are lower than the values published by Yokelson et al. (2009). These are summarized in Table 2.

5 Conclusions

We conducted source characterization and ambient experiments in order to describe and estimate the olive tree branches burning emissions. In addition to NO_x, O₃, CO and CO₂, VOCs such as methanol, acetonitrile, acrolein, benzene, toluenes and xylenes were also detected. The size distributions of the fresh olive tree branches fires had a number mode mobility diameter of 60–80 nm. The aerosol emitted consists of organics, BC, potassium, chloride, nitrate and sulfate. FTIR functional group analysis showed that otBB-OA was composed of 48 % alkane groups, 27 % organic hydroxyl groups, 11 % carboxylic acid groups, 11 % primary amine groups and 4 % carbonyl groups. The O : C ratio of the fresh otBB-OA was estimated 0.29 ± 0.04. The AMS mass spectrum of otBB-OA is characterized by the *m/z*'s 27, 29, 39, 41, 43, 44, 55, 57, 67, 69 and 91. This mass spectrum is quite different from other biomass burning OA mass spectra. After 16 h of exposure to approximately 10⁶ molecules cm⁻³ of OH and 200 ppb O₃ the otBB-OA mass spectrum changes significantly ($\theta = 45^\circ$) with the O : C increasing to 0.4. Part of this change is due to the production of acids.

The levoglucosan to PM₁ O : C ratio was low (0.034–0.043) compared to the most biomass burning sources. This may lead to an underestimation of the biomass burning in the Southern Europe, where burning of olive tree branches is a common agricultural waste management technique in the Mediterranean during the winter and levoglucosan has been used as a wood burning tracer. Applying PMF to ambient AMS measurements we found 3 factors among them the otBB-OA factor contributing 33.7 % to the average organic ambient aerosol. Its mass spectrum is very close to the fresh otBB-OA chamber spectrum. The otBB-OA estimated EF is 3.5 ± 0.2 g kg⁻¹ and is an

Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



important source in the Greek and the Mediterranean Area during the winter months. These emissions should be included in emission inventories and corresponding CTMs.

Supplementary material related to this article is available online at:

<http://www.atmos-chem-phys-discuss.net/13/7223/2013/>

[acpd-13-7223-2013-supplement.zip](#)

Acknowledgements. We thank David Patoulias for his assistance with the TOMAS model and Albert Presto for his help with the pump losses. This research was supported by the European Research Council Project ATMOPACS (Atmospheric Organic Particulate Matter, Air Quality and Climate Change Studies) (Grant Agreement 267099).

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ACPD

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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



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Burning of olive tree branches

E. Kostenidou et al.

Table 1. Conditions for chamber experiments.

Experiment Number	Date	Addition of plastic	Age of olive tree branches	Initial mass concentration ($\mu\text{g m}^{-3}$)	Initial O_3^{a} (ppb)	OH (molecules cm^{-3})
1	14 Dec 2011 Day		Fresh	58	80	NA
2	19 Dec 2011 Day		Fresh/wet	47	230	1.02×10^6
3	10 Feb 2012 Day		2 months old	67	45	0.75×10^6
4	17 Feb 2012 Night	x	2 months old/dry	55	70	0.37×10^6

^aThe ozone presented here is produced during the fire.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Table 2. Emission factors of particle and gas phase species emitted during olive tree branches burning. Units are in g kg^{-1} , for EF_{CO_2} 1500–1700 g kg^{-1} , except for particle number where the EF is in number of particles kg^{-1} .

Species	Emission Factor		
	This work	Sinha et al. (2003)	Yokelson et al. (2009)
Organics	3.22–3.65	–	3.25
Sulfate	0.33–0.37	0.17 ± 0.18	0.047 ± 0.024
Nitrate	0.28	0.16 ± 0.11	0.233 ± 0.056
Chloride	0.04	0.97 ± 1.4	0.509
Black Carbon	0.14–0.16	0.39 ± 0.19	0.541 ± 0.163
NO	0.19–0.21	3.3 ± 0.6	1.733
NO ₂	1.24–1.41	–	4.235
CO	37.53–42.53	68 ± 30	80.18 ± 19.4
<i>m/z</i> 33 (Methanol)*	1.08–1.22	1.2 ± 0.46	2.59 ± 1.27
<i>m/z</i> 42 (Acetonitrile)*	0.10–0.12	–	0.5
<i>m/z</i> 57 (Acrolein)*	0.48–0.54	–	0.096
<i>m/z</i> 79 (Benzene)*	0.17–0.19	0.18 ± 0.09	0.759
<i>m/z</i> 93 (Toluene)*	0.17–0.17	0.13 ± 0.1	–
<i>m/z</i> 107 (Xylenes)*	0.24–0.27	–	–
Particle number (10–500 nm)	$(3.32\text{--}3.77) \times 10^{15}$	$(2.0 \pm 1.5) \times 10^{14\text{a}}$	–

^aSinha et al. (2003) estimated the EF of particles number for particle size 0.1–3 μm .

* Species used for the PTR-MS calibration at the specific *m/z*.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Table 3. Correlations between PMF factors from this work and PMF factors from previous studies.

OOA	Reference	Angle (degrees)
Pittsburgh	Zhang et al. (2005)	25.2
Zurich, summer (OOA-LV)	Lanz et al. (2007)	13.3
Zurich, summer (OOA-SV)	Lanz et al. (2007)	70.8
Zurich, winter	Lanz et al. (2008)	20.8
Pittsburgh (OOA-LV)	Ulbrich et al. (2009)	25.8
Pittsburgh (OOA-SV)	Ulbrich et al. (2009)	40.8
Barcelona (OOA-LV)	Mohr et al. (2012)	49.1
Barcelona (OOA-SV)	Mohr et al. (2012)	52.7
Paris, winter	Crippa et al. (2012)	33.1
HOA	Reference	Angle (degrees)
Pittsburgh	Zhang et al. (2005)	14.4
Zurich, summer	Lanz et al. (2007)	22.0
Zurich, winter	Lanz et al. (2008)	13.5
Pittsburgh	Ulbrich et al. (2009)	19.8
Barcelona	Mohr et al. (2012)	10.8
Paris, winter	Crippa et al. (2012)	25.6
BBOA	Reference	Angle (degrees)
Zurich, summer	Lanz et al. (2007)	49.9
Zurich, winter	Lanz et al. (2008)	38.8
Barcelona	Mohr et al. (2012)	72.8
Paris, winter	Crippa et al. (2012)	49.8
FAME-09	Hildebrandt et al. (2011)	59.9
Olive tree branches burning	This work	10.6

Burning of olive tree branches

E. Kostenidou et al.

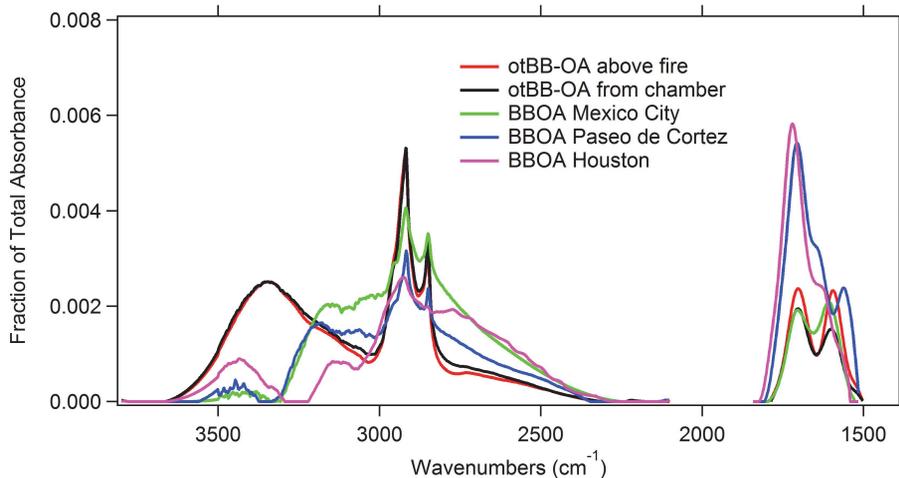


Fig. 1. Normalized FTIR spectra for experiment 2 for the sample taken directly from the olive tree branches fire (red line) and from the chamber the first hour of the experiment (black line). The 2 mass spectra are similar to each other. The otBB-OA spectrum differs a lot from other BBOA FTIR spectra measured in Mexico City, Paseo de Cortez and Houston (Liu et al., 2009; Russell et al., 2009). The correlation coefficients are $R^2 = 0.34$, 0.29 and 0.21 correspondingly.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

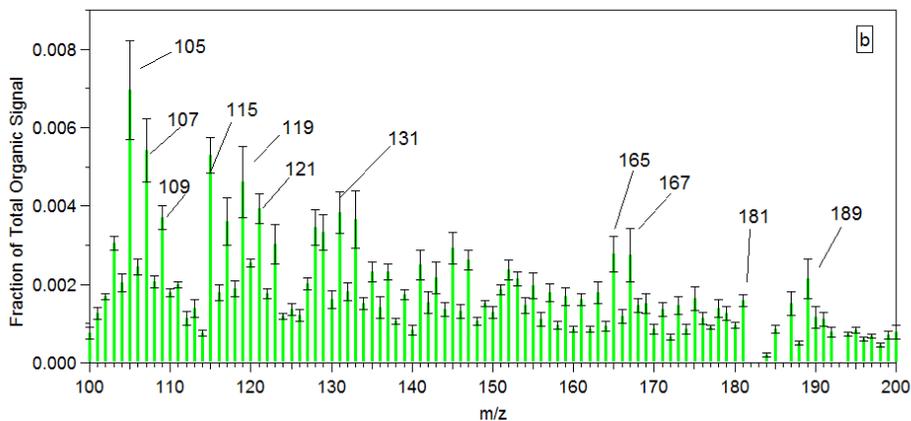
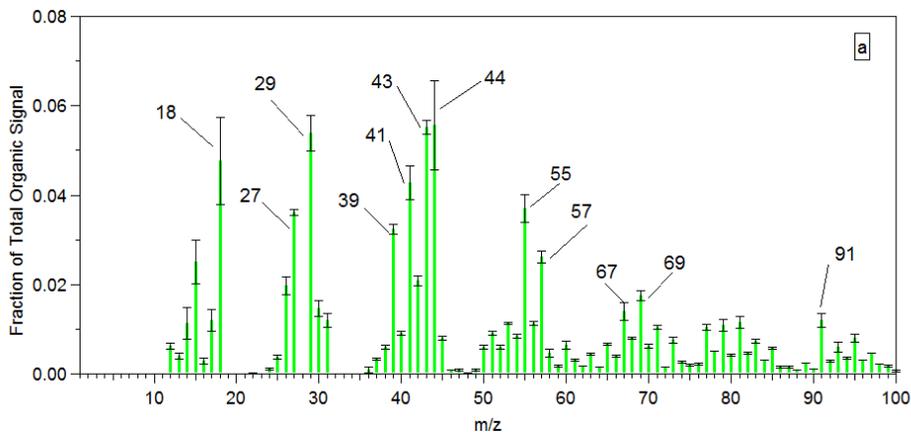


Fig. 2. Average of fresh organic mass spectra of the 4 source characterization experiments: **(a)** the mass spectra from m/z 's 1 to 100 and **(b)** from m/z 's 101 to 200. The black bars correspond to one standard deviation.

Burning of olive tree branches

E. Kostenidou et al.

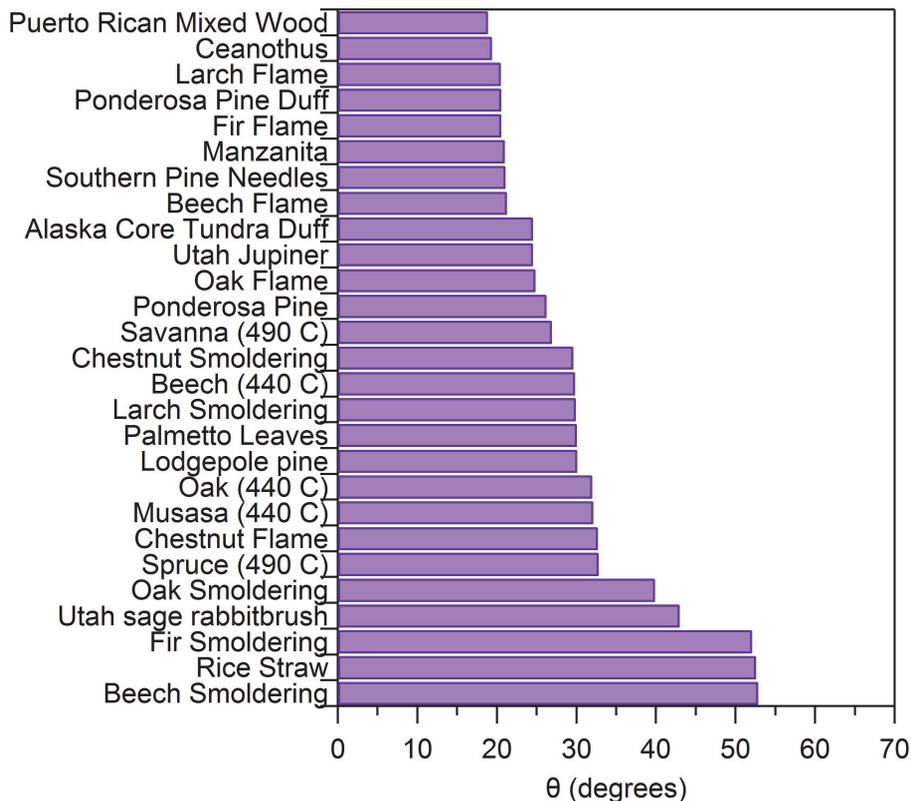


Fig. 3. Angle θ between the organic mass spectrum of the otBB-OA and the BBOA mass spectra. The otBB-OA mass spectrum is quite different from the rest. (Mass spectra source: <http://cires.colorado.edu/jimenez-group/AMSSd/>)

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

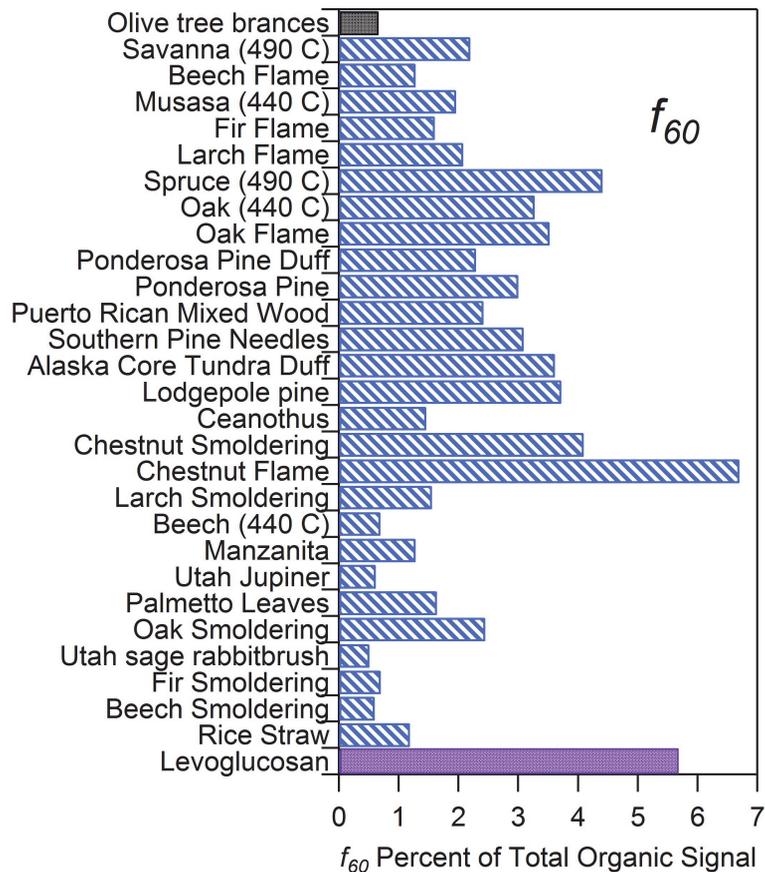


Fig. 4. Contribution of m/z 60 (f_{60}), levoglucosan tracer, to the various BBOA mass spectra. The f_{60} in the olive tree branches burning mass spectrum is only 0.6%.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



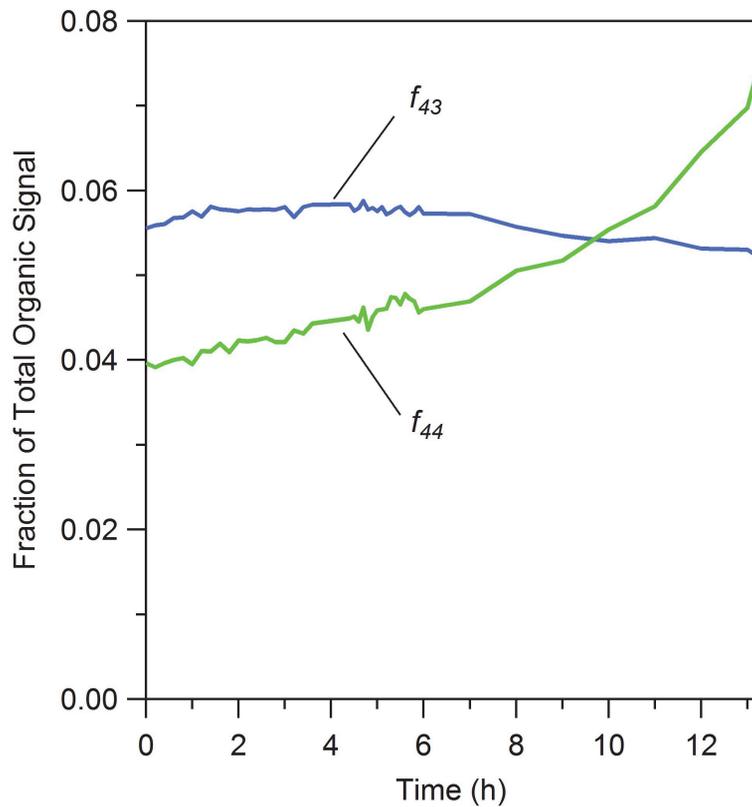


Fig. 5. Time series of f_{44} and f_{43} during the experiment 2. The f_{44} increases through out the experiment, while the f_{43} decreases, which implies that oxidation takes place. This behavior is characteristic of all experiments.

Burning of olive tree branches

E. Kostenidou et al.

Title Page	
Abstract	Introduction
Conclusions	References
Tables	Figures
◀	▶
◀	▶
Back	Close
Full Screen / Esc	
Printer-friendly Version	
Interactive Discussion	



Burning of olive tree branches

E. Kostenidou et al.

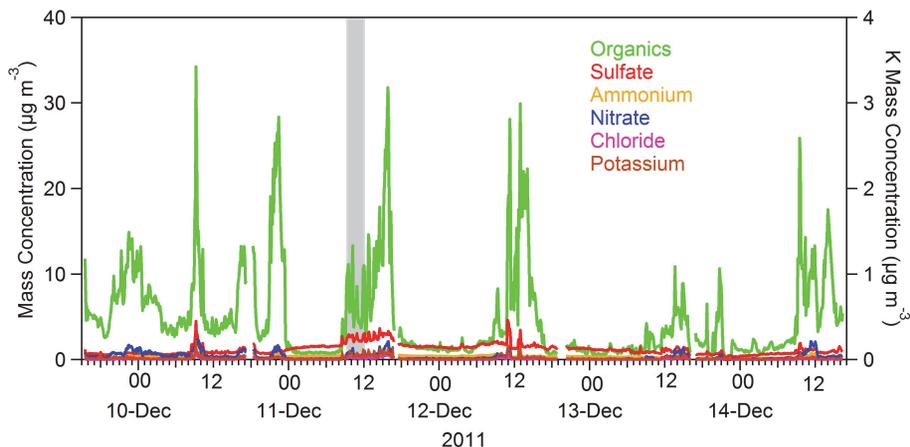


Fig. 7. Example of ambient AMS measurements for 9–14 December 2011. Most of the high OA periods are due to olive tree branches burning in the fields around the sampling area. The mass spectrum during 09:00–12:00 a.m. on 11 December (shaded area) is shown in Fig. 8.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Burning of olive tree branches

E. Kostenidou et al.

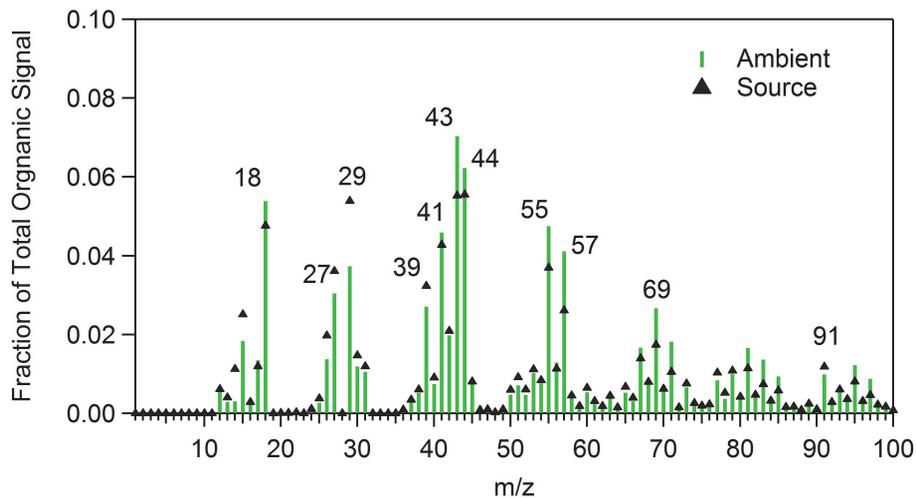


Fig. 8. Comparison between the average mass spectrum from the 4 chamber experiments and the average mass spectrum on 11 December 2012 09:00–12:00 a.m. The angle θ between the two spectra is equal to 13.6 degrees.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Burning of olive tree branches

E. Kostenidou et al.

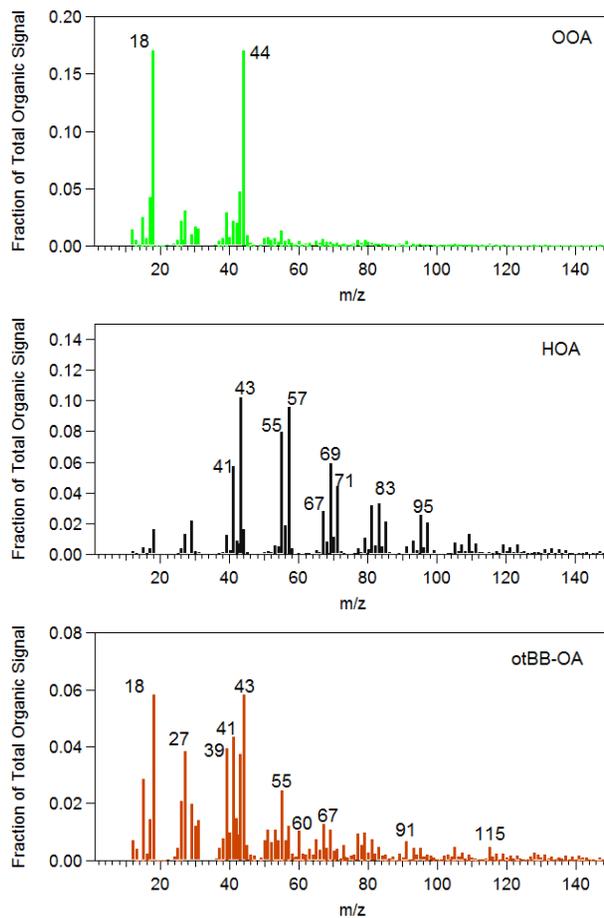


Fig. 9. Mass spectra of OA components from the PMF 3-factor solution.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Burning of olive tree branches

E. Kostenidou et al.

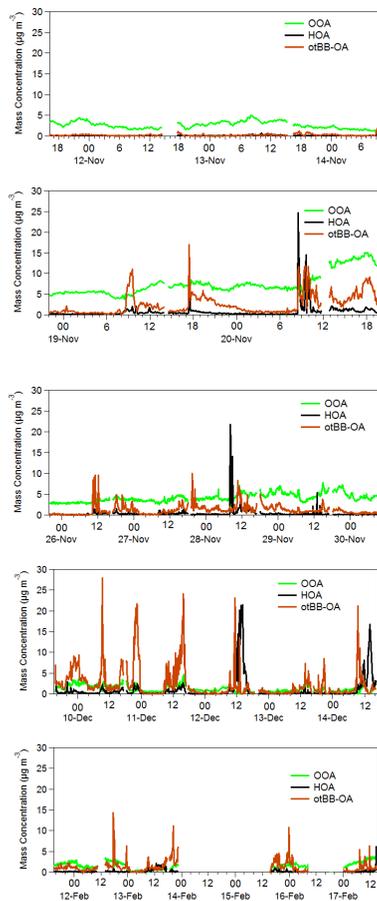


Fig. 10. Time series of the 3 PMF factors for the 20 days, separated in 5 data sets, with an average CE=0.6 applied.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

Burning of olive tree branches

E. Kostenidou et al.

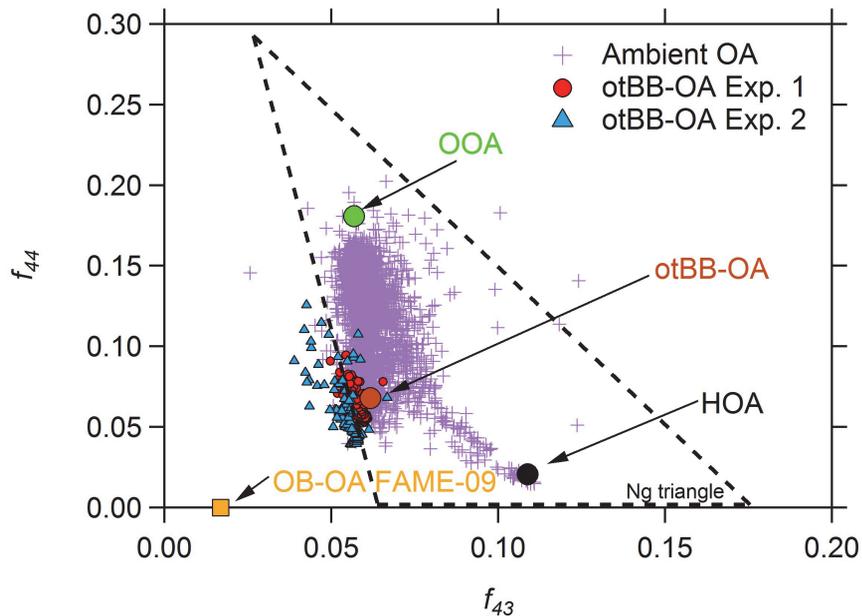


Fig. 11. Ambient and chamber OA data in the Ng et al. (2010) triangle. The ambient OA falls within the triangle. The chamber otBB-OA and the otBB-OA PMF solution are near the bottom left side of the triangle.

Title Page

Abstract

Introduction

Conclusions

References

Tables

Figures

◀

▶

◀

▶

Back

Close

Full Screen / Esc

Printer-friendly Version

Interactive Discussion



Burning of olive tree branches

E. Kostenidou et al.

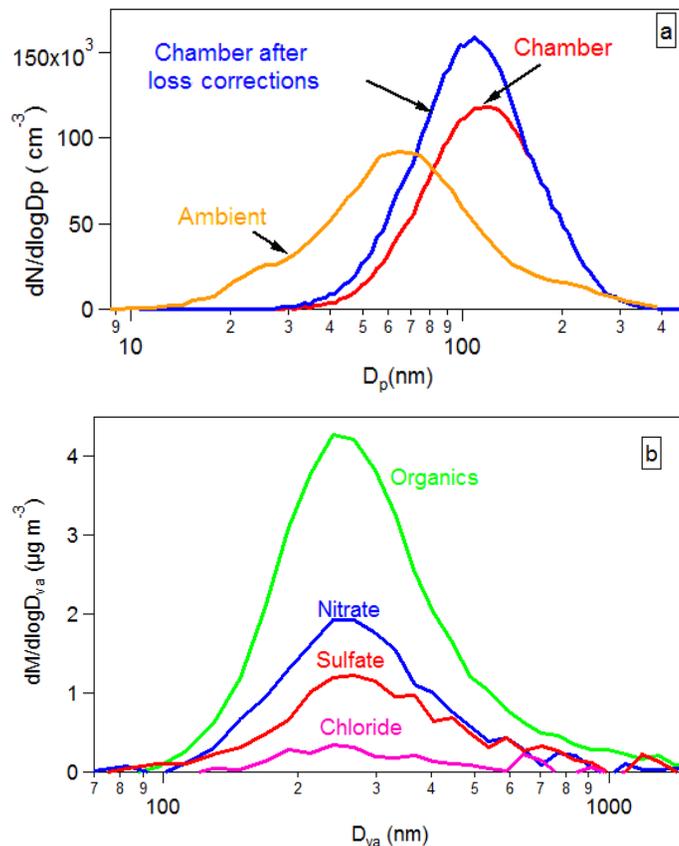


Fig. 12. Size distributions: **(a)** SMPS distributions from experiment 1; the red line is the size distribution as measured in the chamber at $t=0$ (mode diameter ~ 120 nm), the blue line is the reconstructed from the pump losses corrections size distribution for $t=0$ (mode diameter ~ 109 nm) and the orange line is the size distribution during an ambient fire event. **(b)** AMS mass distributions from experiment 1 (mode diameter ~ 240 nm).

Burning of olive tree branches

E. Kostenidou et al.

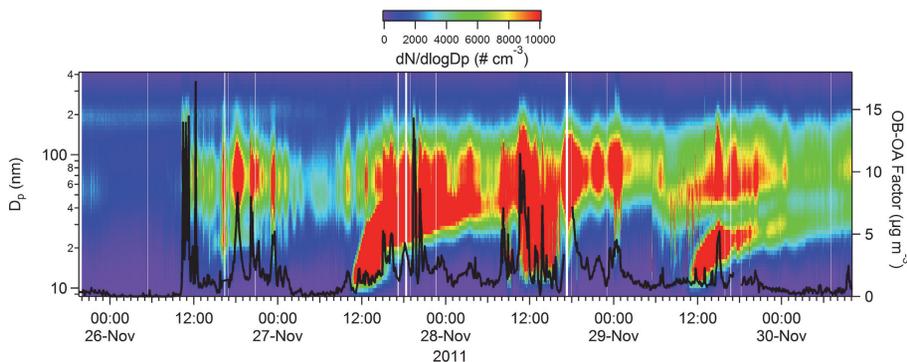


Fig. 13. Size distributions (left axis) plotted with the otBB-OA factor (black solid line, right axis) versus the time from the data set 26–30 November. When the otBB-OA contribution increases the particle diameter is around 60–70 nm.

[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[◀](#)[▶](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)