CHEMICAL SIGNATURE OF BIOMASS BURNING–EMITTED $PM_{2.5}$ AND THE DETECTION OF ITS PRESENCE IN THE AIR BY A RAPID METHOD

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ABSTRACT

Particulate matter (PM) emitted from biomass burning and wildfire has been an air quality concern in affected areas such as dense population centers. Because of regulatory requirements, airborne particles smaller than $2.5 \mu m$ (PM_{2.5}) are of special concern. Controversy has arisen about the source of PM_{2.5}, with smoke from prescribed fire and other biomass burning being a convenient target for elimination by regulators as a means to improve air quality. Because PM_{2.5} may come from several sources, a rapid yet sensitive method is needed to trace PM_{2.5} emitted during biomass burning and to determine if discrimination between sources is feasible. We initiated this research to identify the chemical signature of PM_{2.5} emitted during biomass burning with a rapid multi-element scanning thermal analysis (MESTA) method and to test the hypothesis that this chemical signature can be used to trace its presence in the air. We collected PM_{2.5} samples from the ambient air and experimental burning of pine (*Pinus* spp.) forest biomass and analyzed them with the MESTA method. The MESTA thermograms show that the PM_{2.5} emitted from the prescribed burning of biomass has a characteristic high-temperature-volatile ($<300^{\circ}$ C) component with very low nitrogen (N) and sulfur (S) contents, whereas PM_{2.5} in the ambient air has a characteristic low-temperature-volatile ($<300^{\circ}$ C) component with very high N and S contents. The sharply contrasting MESTA chemical signatures between the experimental biomass burning PM_{2.5} and the ambient air PM_{2.5} provide a promising means to trace and quantify the presence in the air of PM_{2.5} resulting from biomass burning.

Keywords: biomass burning, chemical signature, PM_{2.5}, smoke, wildfire.

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INTRODUCTION

Fire plays an important role in many forest ecosystems (Bååth et al. 1995, Pietikäinen and Fritze 1995, Boerner and Brinkman 2003, Huntzinger 2003, Podur et al. 2003). Periodic fires in these forests have resulted in the evolution of species adapted to and dependent on fire-maintained habitats, such that fire is necessary for maintaining biodiversity (Huntzinger 2003). A hundred years of fire suppression has led to increases in fire-intolerant species (e.g., white fir [Abies concolor] and water oak [Quercus nigra]), increased density of trees, and the accumulation of dead and live fuels that may cause unnaturally intense and severe fires (Pyne 1984, Brose and Wade 2002, Huntzinger 2003).

The use of prescribed burning not only reduces hazardous fuels but also improves wildlife habitat, controls tree pests and diseases, and promotes biodiversity (FDOF 2007). However, one critical environmental concern is the emission of substantial amounts of smoke, including particulate matter (PM) and other pollutants, into the atmosphere. Fire-emitted PM now represents a significant fraction of emission inventories in places where emissions are concentrated (Dennis et al. 2002). Particles with aerodynamic diameter $<2.5~\mu m$ (PM_{2.5}) are of particular concern because of longer suspension time and associated health problems (McMahon 1999, Radojevic and Hassan 1999, Brunekreef and Holgate 2002) and the increased use of prescribed burning as a land management tool (McMahon 1999).

In response to non-attainment of air quality standards in many cities, federal and state environmental agencies are currently considering measures to greatly restrict the use of prescribed burning. For example, in 1994, the Georgia Environmental Protection Division moved to impose summer burning bans on 11 counties surrounding three cities, threatening the biodiversity of tens of thousands of acres of longleaf pineland (Pinus palustris) including the federally endangered red-cockaded woodpecker (Picoides borealis). However, data from the same agency suggested that only a fraction of total PM emissions results from biomass burning (Georgia DNR 1999). Emissions also may be overestimated by the use of burn authorizations to predict area actually burned and unrealistic estimates of fuel loads (Pechan 2005, Robertson and Ostertag 2006). Thus, developing the technology to discriminate between wildfire PM and other sources (e.g., industry, transportation) in non-attainment situations will be critical for guiding sound air quality policy that considers natural resource values and wildfire risk. In

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particular, it is very desirable to have a rapid yet sensitive method for the detection and quantification of biomass burning–emitted PM_{2.5} in the air.

 $PM_{2.5}$ is composed of many different components, including organic carbon, elemental carbon, trace elements, ions, metal oxides, sulfates, nitrates, and ammonium. Carbonaceous matter is a major component of fire-emitted $PM_{2.5}$ and has accounted for 30–60% of the total weight in several studies (Chow et al. 1996, Ward et al. 2004). Carbonaceous matter has been most often categorized into organic, elemental, and carbonate categories (Andreae and Merlet 2001). Other methods of chemical characterization of organic matter in $PM_{2.5}$ have been used but are still not generally applied due to their quite sophisticated and tedious analytical procedures (Oros and Simoneit 2001, Ward et al. 2004).

Sources of atmospheric PM_{2.5} have been determined using various methods. A number of studies used organic carbon and carbon-14 (Cooper et al. 1981, Ward et al. 2006) to determine the source of emissions. The use of radiocarbon can determine whether the emission is biomass or fossil fuel. However, radiocarbon analyses are quite expensive and time consuming. Potassium has been used as a wood smoke tracer, but its emission is fire temperature dependent (Khalil and Rasmussen 2003, Hays et al. 2005) and thus difficult to be quantitatively related to PM_{2.5} emitted during biomass burning. Satellite imagery, GIS, and visibility in addition to air quality data were also used to characterize PM emissions from forest fires (Tanner et al. 2001, Mendoza et al. 2005, Wu et al. 2006). Additionally, organic speciation and combustion biomarkers (e.g., levoglucosan, resin acids, syringols) have been used to determine PM_{2.5} emitted from fires (Simoneit et al. 1999, Oros and Simoneit 2001, Robinson et al. 2006, Ward et al. 2006). Analyses of these compounds include pretreatment steps (e.g., solvent extraction, fractionation and derivatization of gas chromatography-mass spectroscopy) that are time consuming. Accuracy of calibration of levoglucosan to the actual amount of PM emission also remains uncertain. There is no certified U.S. Environmental Protection Agency or National Institute of Safety and Health analytical method for measuring levoglucosan concentrations (Ward et al. 2006). Furthermore, only a few laboratories have experience in measuring levoglucosan, and a study comparing estimates of levoglucosan among participating laboratories showed poor agreement (CV approximately 83%; Ward et al. 2006). A more commonly used analytical method for fire-emitted PM_{2.5} is the thermal-optical method for organic and elemental carbon analysis (Turpin et al. 1990). However, the thermal-optical method gives only the bulk organic and elemental carbon information that may not be detailed enough for tracing purposes.

A multi-element scanning thermal analysis (MESTA) has been developed recently for the characterization of organic matter in solid samples (Hsieh 2007). The MESTA heats up a sample from ambient temperature to 800°C at a given rate and a given atmosphere depending on the application. The volatile component during the heating is combusted at 1,100°C under pure oxygen, and the carbon (C), nitrogen (N), and sulfur (S) contents oxidized and detected quantitatively by their respective detectors. The result of a MESTA is simultaneous C, N, and S thermograms that represent thermal stability of the organic compounds in a sample from ambient to 800°C.

This study was initiated to identify the chemical signature of PM_{2.5} emitted during biomass burning using the MESTA method and to investigate the feasibility of using this chemical signature for the detection and quantification of PM_{2.5} in the air as a result of biomass burning.

EXPERIMENTAL METHODS

PM_{2.5} Sample Collection

PM_{2.5} samples were collected using a PQ 200 PM_{2.5} air sampler (BGI Inc., Waltham, MA) that was programmed to run at a specified time period at a flow rate of 16.7 L/min and with a 47-mm prebaked (850°C for 30 min) and pre-weighed quartz filter (SKC Inc., Eighty Four, PA). After sample collection, the filter was removed from the air sampler and placed in a lined tin can and then into a desiccator prior to reweighing. The quartz filters were weighed using an analytical balance (Mettler Toledo, Columbus, OH) capable of weighing to 1/100th of a milligram. PM_{2.5} mass was determined from the difference between the pre- and postsampling filter weight. The PM_{2.5} concentration (in µg/m³) was calculated using the total volume of air that passed through the filter during the sample collection period. PM_{2.5} filter samples were stored in a freezer prior to the MESTA analysis. Five ambient air PM_{2.5} samples were collected on the roof of a five-story building on the campus of Florida A&M University (FAMU), Tallahassee, Florida, in June and July, 2005. The duration for a sample collection ranged from 18 to 24 hours. Four smoke-impacted air samples were collected on the roof of a five-story campus building at FAMU during 15-20 May 2007 when a smoke alert was issued by the city of Tallahassee on 14 May as the result of the Okefenokee Swamp wildland fire in South Georgia, about 180 km east of Tallahassee, Florida.

Biomass Burning Experiments

Burning experiments were conducted using dead pine wood (*Pinus taeda*), water-soaked dead pine wood, and live pine forest understory vegetation as fuel. Dead pine wood and live understory vegetation were collected from the FAMU Research Farm in Quincy, Florida, and transported to the university main campus in Tallahassee, Florida. Enough dead pine wood was collected to ensure a separate portion could be soaked in water overnight prior to burning.

For the burning experiments, a portable open-flame outdoor fireplace was installed in an open area away from flow of traffic. A portable gas burner with a slotted oven tray placed above it was used inside the fireplace to ensure a constant application of heat for combusting the added fuel. Each experiment started with an approximately 5-kg pile of dead wood, wet wood, or live understory vegetation to be burned. During the burning experiment, one of the fuel types was continuously added to the fireplace to ensure relatively constant weight of the pile throughout the duration of burning. A PM_{2.5} air sampler was placed within 5 m of the fireplace in the downwind direction to collect the smoke PM_{2.5}. The PM_{2.5} sampling duration ranged from 3 to 8 hours in each experiment.

MESTA Analysis

Filter samples were removed from the freezer and placed in a desiccator prior to the MESTA analysis. The whole filter was cut into 16 equal pie-shaped pieces and two to three of them randomly selected for weighing prior to the MESTA analysis. The filtered material was sufficiently adhered to the filter to avoid flaking while being cut. The weight of PM used in the MESTA was estimated from the proportion of the filter fragment to the total weight in the filter, assuming that the PM distribution was relatively even on the filter. Details of the MESTA can be found in Hsieh (2007). Briefly, the sample compartment was programmed to heat from ambient temperature to 800°C at a constant rate of 50°C/min with a 40/60 mixture by volume of extra high purity O, and He gases as the carrier. The volatile fraction of the sample during the analysis was then carried by the carrier gas into a 1,100°C combustion furnace that is continuously fed by 100% extra high purity O₂. The C, N, and S contents of the volatile fraction were oxidized into CO₂, NO₂, and SO₂, respectively, and quantified by the respective CO₂ infrared analyzer, NO₂ chemiluminescence detector, and SO₂ chemiluminescence detector in a serial manner. The gas flow rates in the sample compartment and combustion furnace are 80 mL/min and 350 mL/min, respectively. The heating rate and carrier gas composition of the MESTA were selected for this study according to the procedure of a previous study (Hsieh 2007) that minimizes charring of the sample during the analysis. A PC-based multichannel data logger (National Instrument 6034E, Austin, TX) was used to record the real-time temperature, C, N, and S signals. The C, N, and S contents of a sample were obtained using the calibration curve of standards prepared by reagent-grade cystine, glucose, and elemental sulfur (S₈). After C, N, and S concentrations were obtained in µg/g PM, they were then converted into units of µg/m³ air that takes into account the PM_{2.5} concentration in the air. Temperature of the sample was calibrated using internal standards of elemental sulfur and silver sulfide (Ag₂S). The elemental carbon content of a sample was determined by the fraction of the total carbon volatilized above 550°C using a curve de-convolution software Peakfit (SeaSolve Software 2003) according to the criteria of a previous study by Hsieh and Bugna (2008).

RESULTS AND DISCUSSION

The Tallahassee campus ambient air samples had mean $PM_{2.5}$ concentrations exceeding the National Ambient Air Quality Standard (NAAQS) annual standard (15 μ g/m³) but did not exceed the NAAQS daily standard (35 μ g/m³) for $PM_{2.5}$ (Table 1). The ambient air $PM_{2.5}$ in this study (Figure 1) contained 14–24% total C (excluding inorganic C) compared to the 30–60% observed in other ambient air studies (Chow et al. 1996, Ward et al. 2004). The $PM_{2.5}$ emitted during biomass burning had relatively low elemental carbon concentration (12.8% of total C) compared to those of the ambient air samples (23.7% total C), even though the absolute amount of elemental carbon was much higher in the smoke $PM_{2.5}$ (14 μ g/m³ versus 1.3 μ g/m³). This implies that although the biomass burning produced a lot of elemental carbon, it produced even more organic carbon as $PM_{2.5}$.

Table 1. Concentrations of airborne particles smaller than 2.5 µm (PM₂₅) and its carbon (C), nitrogen (N), sulfur (S), and elemental carbon (EC) contents of the air samples collected in Tallahassee, Florida, in 2005 and 2007.

Sample	μg PM _{2.5} /m³ μg C/m³	µg C/m³	µg N∕m³	µg S/m₃	EC µg C/m³	EC %TC ^a	Bulk 100 × N/C	Bulk 100 × S/C	LTV ^b 100 × N/C	LTV 100 × S/C	HTV ^b 100 × N/C	HTV 100 × S/C
Ambient air (2005) Campus 22–25 Jun	26.5	4.33+0.46	2.07+0.18	2.32+0.17	0.87+0.09	20.0	41.0	20.1	83.5	41.1	13.2	6.4
Campus 26–29 Jul Biomass himing smoke	17.7	2.54 ± 0.15	2.54 ± 0.15 1.63 ± 0.07	1.36 ± 0.09	0.38 ± 0.02	14.8	55.0	20.1	115.5	36.5	18.8	10.0
Dead wood	75.0 ± 13.7	75.0 ± 13.7 67.5 ± 44.9 $2.01\pm0.$	2.01 ± 0.20	Trace	13.51 ± 8.98	20.0	5.6	<0.05	5.6	<0.05	2.1	<0.05
Soaked dead wood	134	129	2.58	Trace	7.21	5.6	1.7	<0.05	1.4	<0.05	2.0	<0.05
Forest understory	187 ± 93.3	168 ± 39.4	3.91 ± 0.30	Trace	21.38 ± 5.00	12.7	2.0	<0.05	2.7	<0.05	1.6	<0.05
Smoke-impacted air (2007) Campus 15–16 Mav.												
smoke-impacted Campus 16-17 May,	61.96	17.97	1.09	0.04	1.44	8.0	5.2	0.1	13.2	0.1	3.5	0.1
less smoke impacted	44.49	5.09	0.94	0.16	09.0	11.8	15.9	1.2	48.6	- -	6.3	1.2
Campus 17-18 May	35.76	3.71	1.74	1.62	0.33	9.0	40.2	16.4	97.2	24.6	14.3	12.7
Campus 18-20 May	30.90	7.58	1.82	1.70	0.56	7.4	20.6	8.4	77.9	14.7	4.5	9.9

^a TC, total carbon. ^b LTV, low-temperature-volatile (<300°C) component; HTV, high-temperature-volatile (>300°C) component.

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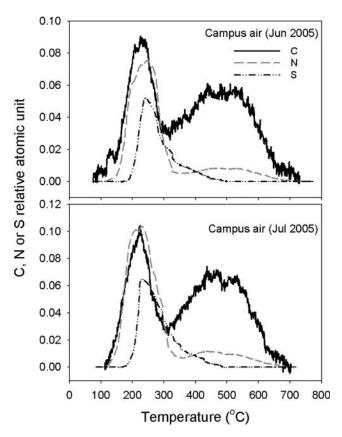


Figure 1. Carbon (C), nitrogen (N), and sulfur (S) thermograms of ambient air $PM_{2.5}$ samples collected in June and July, 2005 on the campus of Florida A&M University, Tallahassee. The C, N, and S contents of a sample were obtained using the calibration curve of standards prepared by reagent-grade cystine, glucose, and elemental sulfur (S $_{\! 8}$). The thermograms were normalized to show the relative atomic ratios of C, N, and S in the PM sample.

The ambient air PM_{2.5} had much higher N and S contents (mean bulk atomic N/C = 0.471 and atomic S/C = 0.192) than the biomass burning $PM_{2.5}$ (mean bulk atomic N/C = 0.021 and atomic S/C < 0.0005). The MESTA thermograms of ambient PM₂₅ (Figure 1) indicate that the PM has two major components with regard to thermal stability: a lowtemperature-volatile (LTV) component decomposed below 300°C and a high-temperature-volatile (HTV) component decomposed above 300°C. The N and S contents of the ambient air PM_{2.5} were concentrated in the LTV component (mean N/C = 1.037 and S/C = 0.351) to a greater degree than in the biomass burning PM_{2.5} (Figure 2). The N concentration of the ambient air PM_{2.5} in its LTV component was > 30 times that of the biomass burning PM₂₅. Most N and S contents in the PM_{2.5} samples are organic, judging from the co-volatile carbon and the temperatures at which they were volatilized (150-500°C). The MESTA shows virtually no inorganic sulfates because their peaks appear upwards of 850°C (Hsieh 2007). Even in ammonium sulfate, the major sulfur peak appears above 850°C, although a small skewed shoulder appears around 650°C. The only inorganic sulfur species that appear in the MESTA are elemental sulfur (peak appears around 98°C) and sulfides (peak appears 450–600°C; Hsieh 2007). Therefore, if a PM sample has sulfate, the majority of it will not be shown in MESTA below 800°C. The sulfur in the MESTA of ambient PM is, therefore, in the organic form, although plenty of sulfate may be present. Inorganic nitrate peaks usually appear in MESTA in the region of 450–550°C, except for ammonium nitrate, which may appear around 200°C (Y.-P. Hsieh, unpublished data).

We found little variation in the chemical signature among the ambient air PM_{2.5} samples between June and July. The variation of the chemical signature within the ambient air (Figure 1) or within the biomass burning samples (Figure 2) was much smaller than between ambient air PM_{2.5} and the biomass burning PM_{2.5} (Figure 1 versus Figure 2). We did not make further chemical identification of compounds in the LTV and HTV components because many more comparisons among the standards and samples need to be done in order to make meaningful interpretations. Also, the objective of this study was not to obtain compound-specific information of the PM, but to identify the chemical signature of the PM and validate its potential use as a fingerprint for tracing purposes. The sharply contrasting MESTA chemical signatures of the biomass burning-emitted PM_{2.5} and ambient air PM_{2.5} indicate that MESTA is a promising tool for sensitively detecting biomass burning-emitted PM2.5 in the air.

The MESTA results on airborne PM_{2.5} samples collected during a smoke alert in Tallahassee due to the wildfire of

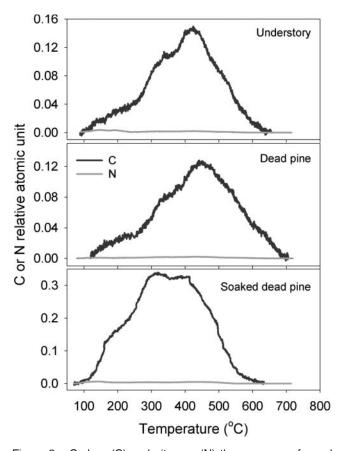


Figure 2. Carbon (C) and nitrogen (N) thermograms of wood smoke $PM_{2.5}$ samples collected during the biomass burning experiments on the campus of Florida A&M University, Tallahassee. The C and N contents of a sample were obtained and thermograms normalized as in Figure 1.

the Okefenokee Swamp in South Georgia, approximately 180 km east of Tallahassee, are shown in Figure 3. The PM_{2.5} in smoke-impacted air collected on 15–16 May 2007 clearly showed the signature of biomass-burning PM, which had very low N and S contents in the LTV component. The biomass-burning signature of PM gradually faded away in the 16-17 May sample and was almost completely gone in the 17-18 May and 18-20 May samples (Figure 3). The PM_{2.5} in the impacted air reached 61.9 μ g/m³ on 15–16 May and then declined to 30.9 µg/m³ on 18–20 May. The chemical signature of PM_{2.5} from the Okefenokee Swamp wildfire appeared to be very similar to that produced in our biomass burning experiments. The MESTA thermograms of the sequential PM_{2.5} samples in the 16–17 May (Figure 3B), 17-18 May (Figure 3C), and 18-20 May (Figure 3D) samples clearly show the gradual fading of the PM chemical signature from biomass burning to ambient air in Tallahassee. Assuming the MESTA chemical signature of ambient air PM_{2.5} in Tallahassee and that of biomass burning-emitted PM_{2.5} represent two end members of PM sources in the impacted air in 15-20 May, a simple linear model using N and S contents in the LTV and HTV components estimated

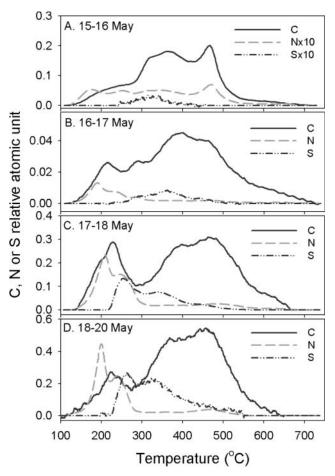


Figure 3. Carbon (C), nitrogen (N), and sulfur (S) thermograms of the impacted air $PM_{2.5}$ samples of Tallahassee, Florida, collected after a smoke alert issued by the city on 14 May 2007. The C, N, and S contents of a sample were obtained and thermograms normalized as in Figure 1. The N and S thermograms in graph A were magnified $10\times$ compared to those in graphs B, C, and D.

that the wildfire-emitted PM composed approximately 90% of the 15–16 May sample, 50% of the 16–17 May sample, 10% of the 17–18 May sample, and 20% of the 18–20 May sample. These estimates are based on the assumption that the PM from the Okefenokee Fire is chemically similar to that measured during our burning experiments, although we did not verify this assumption by measuring PM known to come only from that fire. However, the above calculation serves as an example to show how the MESTA chemical signature could be used to de-mix the sources of PM in the air.

MANAGEMENT IMPLICATIONS

This study shows that MESTA is a sensitive and convenient way to characterize the organic matter of PM_{2.5} in the air in terms of the C, N, and S thermograms. The MESTA chemical signature provides a convenient means to differentiate PM_{2.5} emitted during biomass burning from that of the ambient air. The specific results of this study, however, cannot be generalized to all fires and ambient air because only limited sources of biomass burning and ambient air are investigated. Nevertheless, this study shows that the MESTA is a promising tool for quantifying the contribution of biomass burning-emitted PM_{2.5} in the air, which is needed for research and regulatory purposes with regard to the effect of prescribed fires and wildfires on air quality. We suggest that future studies be conducted that use other forms of biomass (e.g., grasses, deciduous shrubs) and ambient air from other environments before this approach is more broadly applied. We also recommend that this approach be compared directly with other source apportionment approaches.

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