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Method development for the analysis of particle phase substituted methoxy phenols and aromatic acids from biomass burning using capillary electrophoresis/electrospray ionization mass spectrometry (CE/ESI-MS)

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Abstract

A method is developed for the determination of substituted methoxy phenols and aromatic acids in biomass burning aerosol using capillary electrophoresis (CE) coupled to an electrospray ionization mass spectrometer. Background electrolytes (BGEs) containing ammonium acetate, ammonium hydroxide and 10% (v/v) methanol at pH 9.1 and ammonium hydroxide at pH 11 are investigated for their suitability. A good linearity is found for all analytes in the range of 1–50 μM for the ammonium acetate based BGE and 1–40 μM for the ammonium hydroxide BGE. The detection limit ranged from 0.1 to 1.0 μM for the ammonium acetate based BGE and 0.3 to 0.7 μM for the ammonium hydroxide BGE. The relative standard deviation (R.S.D.) is typically less than 0.5% (ammonium acetate based BGE) and 4.2% (ammonium hydroxide BGE) for the migration time and 3–9% (ammonium acetate based BGE) and 2.5–8% (ammonium hydroxide BGE) for the peak area ($n = 5$). The analytical time was less than 10 min for both methods. The proposed methods are fast, sensitive and quantitative and can be applied to the analysis of complex biomass burning aerosol samples without complex pre-treatment. The results from the analysis of real biomass burning samples demonstrate the suitability of the proposed methods to the analysis of low concentration water soluble organic carbon (WSOC) in biomass burning samples. The fast analytical time and high sensitivity of the proposed methods enables the analysis of a large number of size segregated impactor samples from biomass burning aerosols.

Keywords: Biomass burning; Aerosols; Methoxy phenols; Aromatic acids; Phenolic compounds

1. Introduction

Biomass burning contributes significantly to the global aerosol burden and it is an important source of organic aerosols. It primarily consists of decomposed lignin, hemicellulose and cellulose products which include various substituted phenols [1–4]. Gas chromatography–mass spectrometry (GC–MS) previous derivatization is widely used for the analysis of methoxy and dimethoxy phenols from biomass burning aerosol [5–7]. The sample preparation for GC–MS analysis is time consuming and it requires a relatively

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large quantity of the sample which is often difficult for atmospheric aerosol samples. Solid-phase extraction (SPE) and solid-phase micro extraction (SPME) has been extensively used for the pre-concentration of low concentration phenolic compounds in water samples. Rodriguez et al. [8] summarized the current status of SPE and SPME for the analysis of phenolic compounds in water samples. SPME (e.g. [9]) is especially an attractive tool for the pre-concentration of low volume samples such as atmospheric aerosols as it is fast, solvent free and very sensitive. However, only a few applications of SPME to atmospheric samples are reported [10,11] and extensive testing is still necessary for the application of SPME to atmospheric samples.

Capillary electrophoresis (CE) requires very small sample volumes and no sample pre-treatment is necessary. These advantage together with its very high separation efficiency, speed and simplicity make CE a highly attractive analytical technique for the analysis of atmospheric aerosol samples. These advantages also give greater flexibility for the choice of aerosol sampling method and duration. CE with a UV indirect detection method has been successfully applied to the analysis of low concentration atmospheric aerosol or rain samples, especially for the determination of carboxylic acids [12–18]. Although it is relatively new to the analysis of atmospheric samples, the high separation efficiency of CE coupled with sensitivity and selectivity of electrospray ionization mass spectrometry (CE/ESI-MS) is expected be a powerful tool for the speciation and identification of organic compounds in atmospheric particles.

Some work has shown a successful separation of substituted phenols in various matrixes using a CZE [19–22] or MEKC [23,24] with direct UV detection. CE/ESI-MS is successfully applied to separate eighteen chlorophenols by Jauregui et al. [25] using 5 mM diethylmalonic acid at pH 7.25. Due to high pK_a values of substituted phenols, background electrolytes (BGEs) often utilize pH >7. Phosphate or borate with organic solvents is commonly used as buffer system for the separation of substituted phenols [20–22,24]. For CE/ESI-MS system, borate or phosphate buffer systems are incompatible due to their low volatilities. Therefore, ammonium acetate or hydroxide based buffer systems compatible with CE/ESI-MS are evaluated here for the separation and quantification of substituted phenols. In this work, linearity and reproducibility of MS compatible buffer systems are investigated under optimized conditions. The methods evaluated here are fast and quantitative and they can be applied to real atmospheric aerosol samples.

2. Experimental

2.1. Chemicals

Chemicals used in this study were obtained from the following suppliers: vanillic acid (≥97.0%), cinnamic acid (≥99.0%), coniferyl aldehyde (98.0%), 3,5-dimethoxy-4-hydroxyacetophenone (97.0%), syringol (99.0%), 4-hydroxycinnamic acid (98.0%), 3-hydroxy-4-methoxybenzoic acid (97.0%) and eugenol (99.0%) from Aldrich (Munich, Germany); ferulic acid (≥98.0%), sinapic acid (97.0%), vanillin (98.0%), ammonium acetate (≥99.0%), ammonium hydroxide (BioChemika Ultra grade), methanol (≥99.8%), isobutanol (≥99.5%), acetonitrile (≥99.5%) and methyl red (ACS grade) were purchased from Fluka (Munich, Germany); homovanillic acid (98.0%) was obtained from Sigma (Munich, Germany); hydrochloric acid (reagent grade) was purchased from Riedel-de Haën (Munich, Germany).

A 5 mM stock solution of mixed substituted methoxy phenols and aromatic acids was prepared by dissolving appropriate quantities in acetonitrile. The 0.5, 1, 5, 10, 25 and 50 μM standard solutions were prepared by dilution of stock solution with Milli-Q water (Millipore, Bedford, USA). The stock solution and standard solutions were stored at 4 °C in darkness.

Ammonium acetate based BGE solution was prepared in a volumetric flask by diluting appropriate amount of 1 M ammonium acetate stock solution into Milli-Q water, adding an appropriate volume of methanol, pH was then adjusted by adding ammonium hydroxide. Ammonium hydroxide solution from the manufacturer was used as BGE without any modification. The concentration of ammonium hydroxide in the BGE solution was determined by titrating with 1 M HCl and methyl red. The concentration of ammonium hydroxide in BGE was 0.94 M. Ammonium acetate buffer solution was prepared weekly and the pHs of both BGEs were checked periodically.

2.2. Instrumentation

2.2.1. CE system

CZE separations were carried out on Agilent ^{3D}CE instrument (Agilent, Waldbronn, Germany). The fused silica capillary from Chromatographie Service GmBH (Langerwehe, Germany) were 50 μm i.d. (365 μm o.d.) × 73 cm long for the ammonium acetate based BGE and 50 μm i.d. (365 μm o.d.) × 55 cm for the ammonium hydroxide BGE. The separation was carried out at −30 kV at 25 °C. Sample injection was carried out by hydrodynamic injection (50 mbar for 10 s). A new capillary was rinsed with methanol for 5 min, Milli-Q water 5 min, 0.1 M HCl for 5 min, 0.1 M NaOH for 5 min, 1 M NaOH for 5 min, Milli-Q water for 10 min and finally BGE solution for 10 min. As a daily routine, the capillary was flushed with Milli-Q water for 10 min and BGE solution for 10 min before the first run. In between runs, the capillary was flushed with Milli-Q water for 2 min and BGE for 2 min. At the end of the day, the capillary was washed with BGE for 5 min, 0.1 M NaOH for 2 min and finally Milli-Q water for 10 min.

2.2.2. MS system

The detector was a Bruker Esquire 3000 plus ion trap mass spectrometer (Bruker-Daltonics, Bremen, Germany) equipped with an electrospray ionization source (Bruker-Daltonics, Bremen, Germany). The electrospray was operated at the negative mode. Nitrogen was used as drying gas at a 250 °C and a flow-rate of 4 l/min. Nitrogen sheath gas for electrospray was supplied at 3 psi. The sheath liquid (water 50%/*iso*-propanol 50%, v/v) was delivered to electrospray by a micro syringe pump (Cole-Parmer, USA) at 3 μl/min. The voltage set for the MS capillary was 4.5 kV and electrospray current was kept under 300 nA. End plate off-set was fixed at −0.5 kV. Scanning mass range was from *m*/*z* 50–500 with scanning speed of 13,000 *m*/*z* per second, maximum accumulation time of 30.00 ms and performing 12 microscans.

3. Results and discussion

3.1. Separation of substituted phenols

3.1.1. Ammonium acetate based BGE

The separation and selectivity of substituted phenols can be optimized by the concentration of BGE, pH and organic solvents. In order to ensure the compatibility of BGE to the MS interface, the combination of ammonium acetate and ammonium hydroxide is chosen as a BGE. In order to have better ionization of less acidic substituted methoxy phenols, a basic buffer is employed throughout the study. The effect of ammonium acetate concentration on the separation was tested with 10, 15, 20, 25 and 30 mM at pH 9.1. Lower buffer concentration can increase the electroosmotic flow (EOF), hence reducing the analytical time. On the other hand, insufficient buffering capacity of lower concentration BGE can cause detrimental migration time shifts. Higher concentration buffer can improve the peak shape but it increases the analytical time and may produce large amount of heat due to the Joule effect. The resolution of 10 mM ammonium acetate was found unsatisfactory due to poor separation between coniferyl aldehyde and 3,5-dimethoxy-4-hydroxyacetophenone, and vanillin and vanillic acid. Generally speaking, the increase of the ammonium acetate concentration leads to the decrease in sensitivity (S/N). Ammonium acetate concentration between 15 and 25 mM was found to be a suitable for the analysis since it gave a sufficient resolution and maximum intensity at adequate capillary current (<25 μA). Considering the resolution, selectivity, capillary current and analytical time, ammonium acetate concentration of 20 mM was chosen.

The pH value of BGE is an important parameter for CZE separation. Since the pK_a of substituted methoxy phenol compounds without a carboxylic group are above 7, the effect of pH was investigated with 20 mM ammonium acetate buffer at 8.4, 8.8, 9.0, 9.1, 9.2 and 9.3. A pH value of 9.1 gave the best resolution and selectivity. Thus pH 9.1 is used in the subsequent studies.

The resolution and analytical time were further optimized by adding methanol (0, 5, 10, 15 and 20%, v/v). Higher methanol concentration reduces the EOF speed, hence increases the analytical time significantly. A 5% increase in methanol concentration increased the analytical time approximately by 14%. The separations between coniferyl aldehyde and 3,5-dimethoxy-4-hydroxyacetophenone, vanillin and sinapic acid, and vanillic acid and cinnamic acid were not satisfactory below 10% methanol. Therefore, a methanol content of 10% (v/v) is chosen considering the resolution and analytical time.

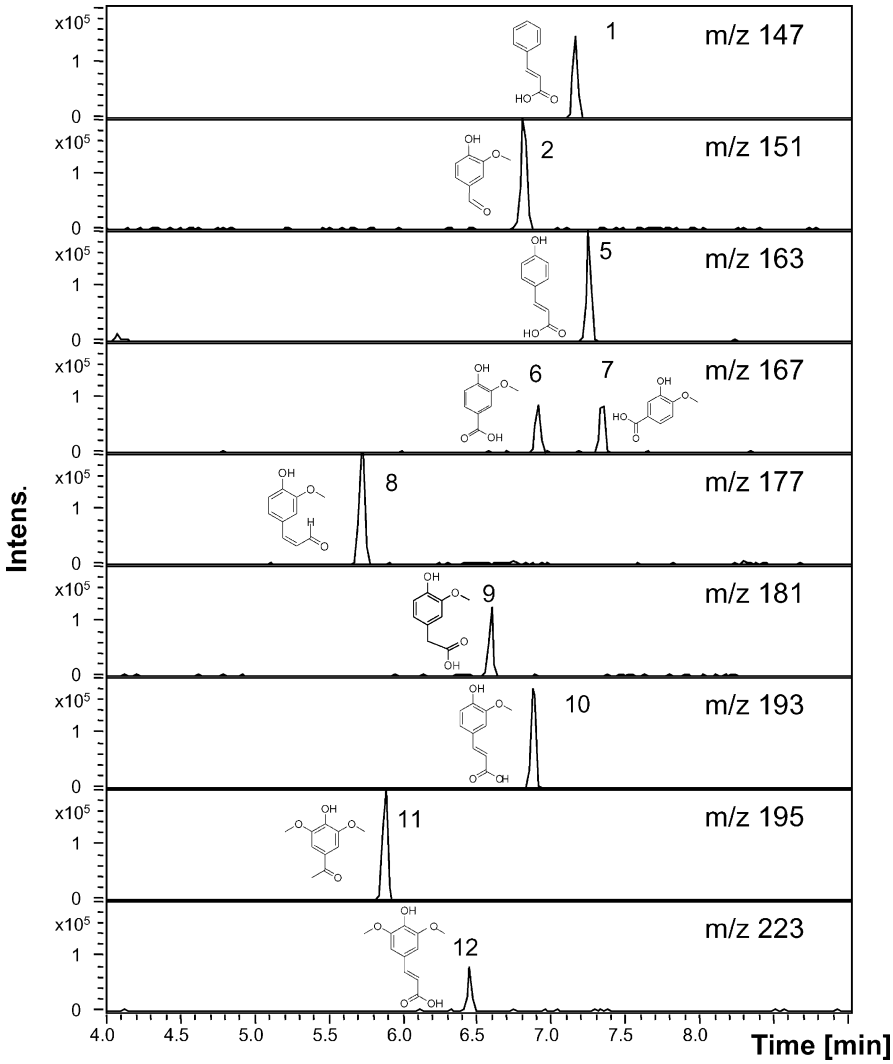

Fig. 1. A typical CE–MS electropherogram of substituted methoxy phenols and aromatic acids using ammonium acetate based BGE under optimized condition. Capillary: 75 μm i.d. × 73 cm; BGE: 20 mM ammonium acetate-10% methanol (pH 9.1); separation voltage 30 kV; capillary current 21 μA; injection: 50 hPa for 10 s; MS polarity: negative; peak identification as in Table 1 (each 20 μM).

Fig. 1 shows a typical electropherogram of a standard mixture containing 12 substituted phenols under optimized condition. Eugenol and syringol eluted with the EOF (4.1 min) and were not quantified. Compounds containing an aldehyde group except vanillin migrate first and compounds without a methoxy group migrate towards the end except 3-hydroxy-4-methoxy benzoic acid. However, no clear relationship was found between migration order, molecular weight, the size of molecules or the position of carboxylic or methoxy group (Table 1). Fig. 2 shows the background subtracted mass spectra of detected compounds using ammonium acetate BGE. All compounds were detected as $[M - H]^-$ and no adduct with acetate $[M + 60 - H]^-$ or clusters were observed. Table 2 lists the reproducibility and detection limit for the compounds using ammonium acetate as a BGE. Acceptably good linear calibration curves ($R^2 > 0.992$) were obtained for standard solutions in the concentration range of 1–50 μM. The relative standard deviations

Table 1
Molecular weight and diagnostic ions of substituted phenols in this study

Peak no.	Compound	M_r	MS diagnostic ion $[M - H]^-$
1	Cinnamic acid	148	147
2	Vanillin	152	151
3	Syringol	154	153
4	Eugenol	164	163
5	4-Hydroxycinnamic acid	164	163
6	Vanillic acid	168	167
7	3-Hydroxy-4-methoxy benzoic acid	168	167
8	Coniferyl aldehyde	178	177
9	Homovanillic acid	182	181
10	Ferulic acid	194	193
11	3,5-Dimethoxy-4-hydroxyacetophenone	196	195
12	Sinapic acid	224	223

($n = 5$) of the proposed method for all detected compounds fell below 0.4% for the migration time and 9% for the peak area. Detection limits (S/N = 3) below 1 μM were achieved for all detected analytes.

3.1.2. Ammonium hydroxide BGE

A subsequent experiment was carried out to study a possible application of ammonium hydroxide as a BGE in order to separate compounds with higher pK_a values such as eugenol and syringol. The major problem encountered when transferring CZE–UV methods capable separating these compounds to CZE/EIS-MS methods is incompatibility of BGEs as well as BGE modifiers to the CE–MS interface. Therefore, the purpose of this part of the study is to develop a fast separation method for the determination of compounds with higher pK_a.

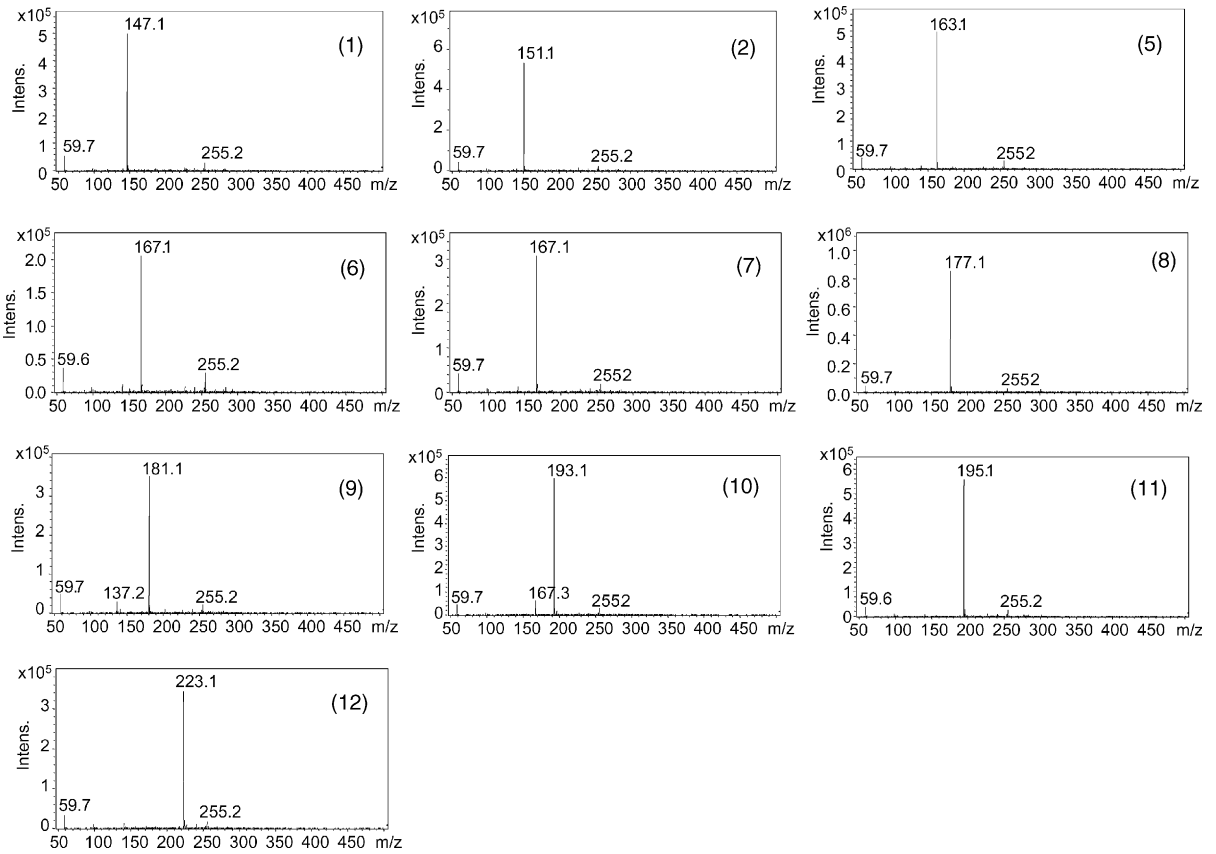

Fig. 2. A typical background subtracted mass spectra of substituted methoxy phenols and aromatic acids with ammonium acetate based BGE. A number at the right corner of each mass spectrum corresponds to a peak number.

Table 2
Reproducibility and detection limit for ammonium acetate based BGE

Peak no.	Compound	Reproducibility		Detection limit (μM)
		Migration time, R.S.D. (%), $n = 5$	Peak area, R.S.D. (%), $n = 5$	
1	Cinnamic acid	0.3	6.4	0.1
2	Vanillin	0.3	3.6	0.5
5	4-Hydroxycinnamic acid	0.3	8.8	0.4
6	Vanillic acid	0.3	6.8	1.0
7	3-Hydroxy-4-methoxy benzoic acid	0.4	8.1	1.0
8	Coniferyl aldehyde	0.3	4.9	0.1
9	Homovanillic acid	0.3	6.5	0.8
10	Ferulic acid	0.4	5.0	0.2
11	3,5-Dimethoxy-4-hydroxyacetophenone	0.3	3.8	0.3
12	Sinapic acid	0.4	6.3	0.6

Conditions as in Fig. 1; detection limit: S/N = 3.

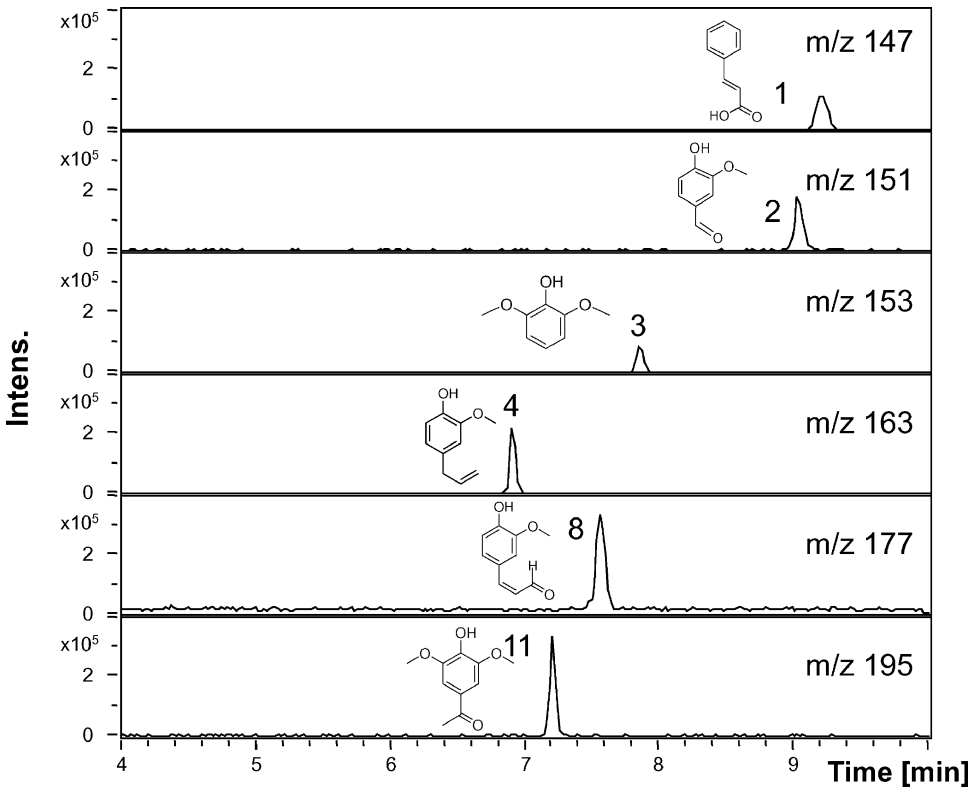

Fig. 3. A typical CE/MS electropherogram of substituted methoxy phenols and aromatic acid using ammonium hydroxide BGE under optimized condition. Capillary: 75 μm i.d. × 55 cm; BGE: 1 M ammonium hydroxide (pH 11); separation voltage 30 kV; capillary current 14 μA; injection: 50 hPa for 10 s; MS polarity: negative; peak identification as in Table 1 (each 20 μM).

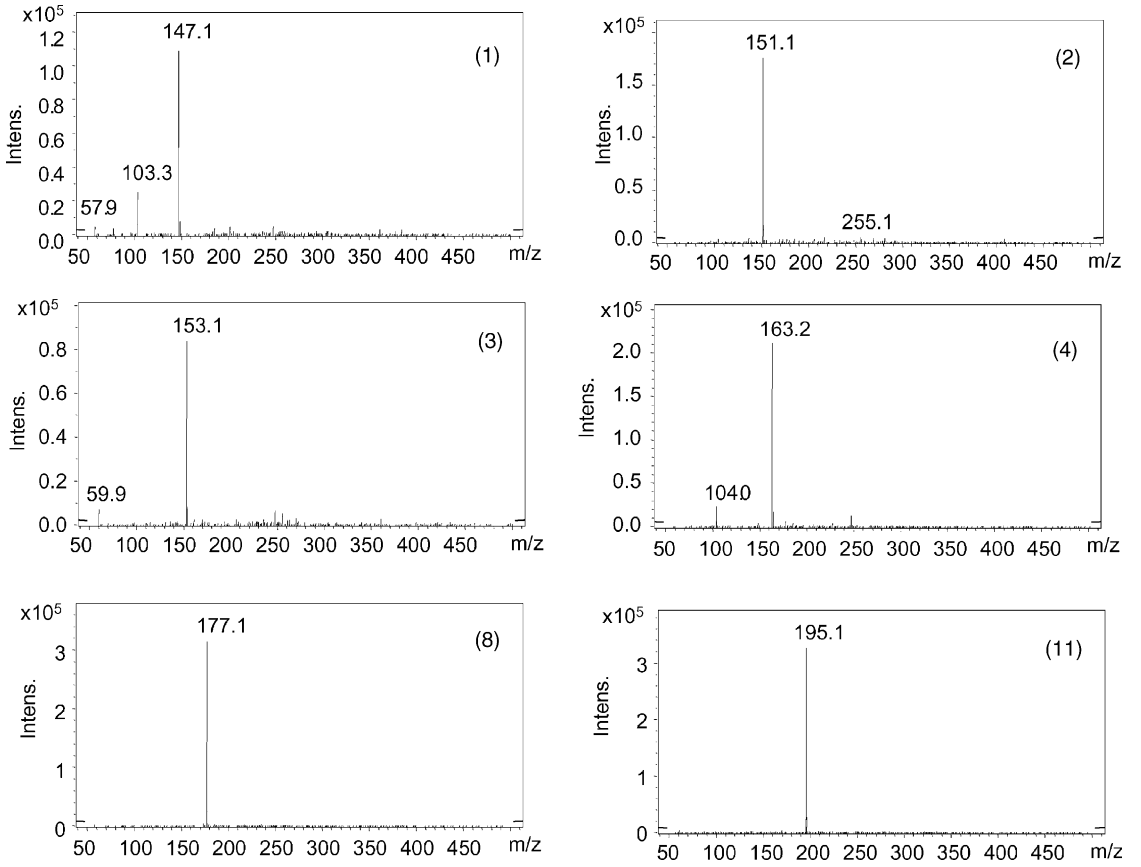

Fig. 4. A typical background subtracted mass spectra of substituted methoxy phenols and aromatic acid with ammonium hydroxide BGE. A number at the right corner of each mass spectrum corresponds to a peak number.

Ammonium hydroxide was chosen as the BGE because of its high pH (∼11) and volatility. Further studies found out that the separation can be performed at very high concentration without suffering from harmful Joule heating effects. Therefore, 0.94 M ammonium hydroxide was chosen for this study. It was found that higher pH of ammonium hydroxide BGE increased the degree of dissociation

Table 3
Reproducibility and detection limit for ammonium hydroxide BGE

Peak no.	Compound	Reproducibility		Detection limit (μM)
		Migration time, R.S.D. (%), $n = 5$	Peak area, R.S.D. (%), $n = 5$	
1	Cinnamic acid	4.2	5.3	0.3
2	Vanillin	4.1	7.6	0.5
3	Syringol	3.1	2.5	0.6
4	Eugenol	2.6	5.5	0.4
8	Coniferyl aldehyde	2.9	6.7	0.3
11	3,5-Dimethoxy-4-hydroxyacetophenone	2.7	3.8	0.7

Conditions as in Fig. 2; detection limit: S/N = 3.

for phenolic OH, hence the effective mobility and the analytical time. Therefore, the separation capillary was shortened to 55 cm for faster migration of the analytes.

Fig. 3 gives an example of an electropherogram for a standard solution containing 12 substituted phenols under optimized condition for ammonium hydroxide BGE. The EOF value of this BGE was 3.4 min. With this BGE, eugenol and syringol which eluted with EOF using ammonium acetate based BGE were clearly separated and detected. However, sinapic acid, homovanilic acid, ferulic acid, vanillic acid, 3-hydroxy-4-methoxy benzoic acid and 4-hydroxycinanamic acid were not detected within a reasonable analytical time. Overall, 6 out of 12 analytes migrated and were detected. Analytes migrated in the order of molecular weight except eugenol. Fig. 4 gives the background subtracted mass spectra of detected compounds with ammonium hydroxide BGE. No adducts of ammonium hydroxide were observed and all compounds were detected as $[M - H]^-$.

Table 3 lists the reproducibility and detection limit of the detected analytes for this BGE. Acceptably linear calibration curves ($R^2 > 0.995$) were obtained for the migrated compounds in the rage of 1–40 μM. The detection limits were slightly higher than that of corresponding detection limits for ammonium acetate based BGE due to noisier baselines for this method. The relative standard deviations of this method for detected compounds were below 5% for the migration time and 8% for the peak area. The higher reproducibility of this method compared to the ammonium acetate based BGE may be explained by the lower variability of run to run pH and BGE concentration due to much

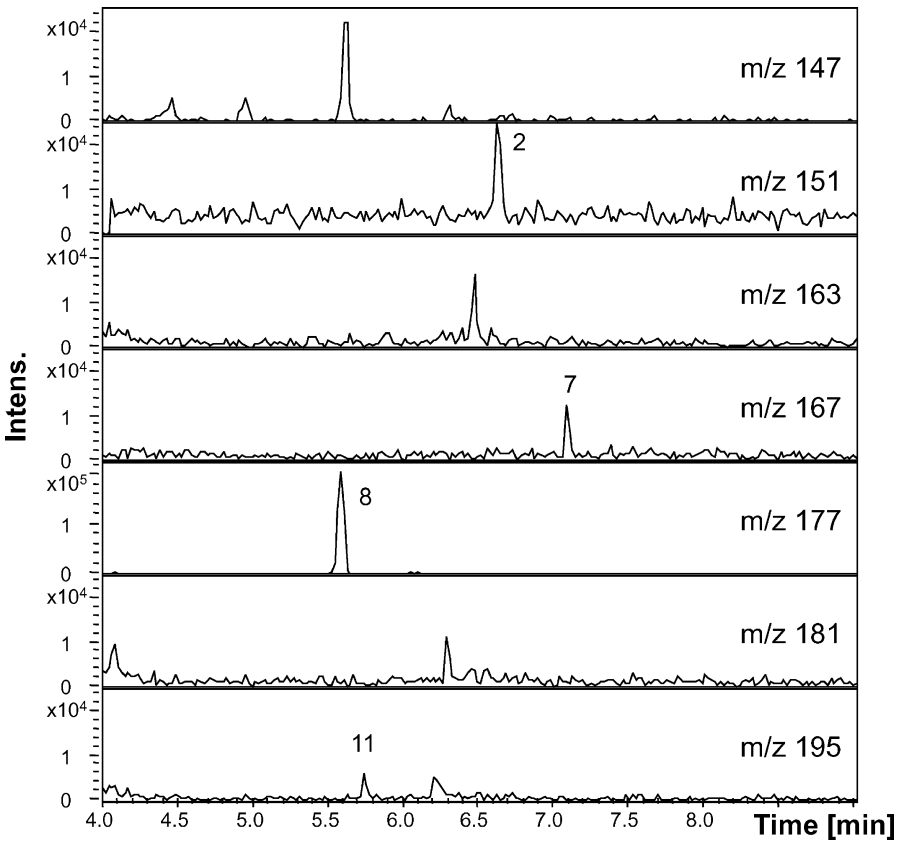

Fig. 5. An example of a biomass burning aerosol sample (pine wood) analyzed by the proposed method. Conditions as in Fig. 1; peak identification as in Table 1.

higher concentration of BGE. It was found that rigorous capillary conditioning was essential to minimize run to run shifts in migration time due to its high BGE concentration.

3.2. Analysis of biomass burning particles

The proposed methods were applied to determine the concentration of substituted phenols in a filter extract from a biomass burning aerosol sample taken during a field campaign EFEU (Impact of Vegetation Fires on the Composition and Circulation of the Atmosphere; http://projects.tropos.de:8088/afo2000g3/EFEU_dateien/efeu.html). A detail characterization of aerosol chemical composition from various wood types is under way. The wood combustion was carried out in a container designed to study biomass burning under controlled conditions. Produced biomass burning aerosols were drawn to the chimney located above the fire place then to an insulated sampling tube. The samples were diluted up to 20 times using synthesized air in a dilution tunnel located after the sampling tube. A five stage Berner type impactor was attached at the end of the dilution tunnel for size segregated particle sampling. The resulting substrates were extracted in 1.5 ml of Milli-Q water for the analysis of inorganic ions with ion chromatography, dicarboxylic acids with CE–UV indirect method and water soluble organic carbon (WSOC) with CE–MS. Figs. 5 and 6 show typical electropherograms from biomass burning aerosol from pine wood at the impactor stage 2 (140–420 nm, 50% cut-off) for the ammonium acetate based BGE and the ammonium hydroxide BGE, respectively. No interference from the matrix was observed for both BGEs. Peaks were identified by their migration time, *m*/*z* and standard spiking.

The concentrations for the detected compounds in the same filter extract using the ammonium acetate based BGE and the ammonium hydroxide BGE are summarized in Table 4. The determined concentrations from both methods are in reasonable agreement. A

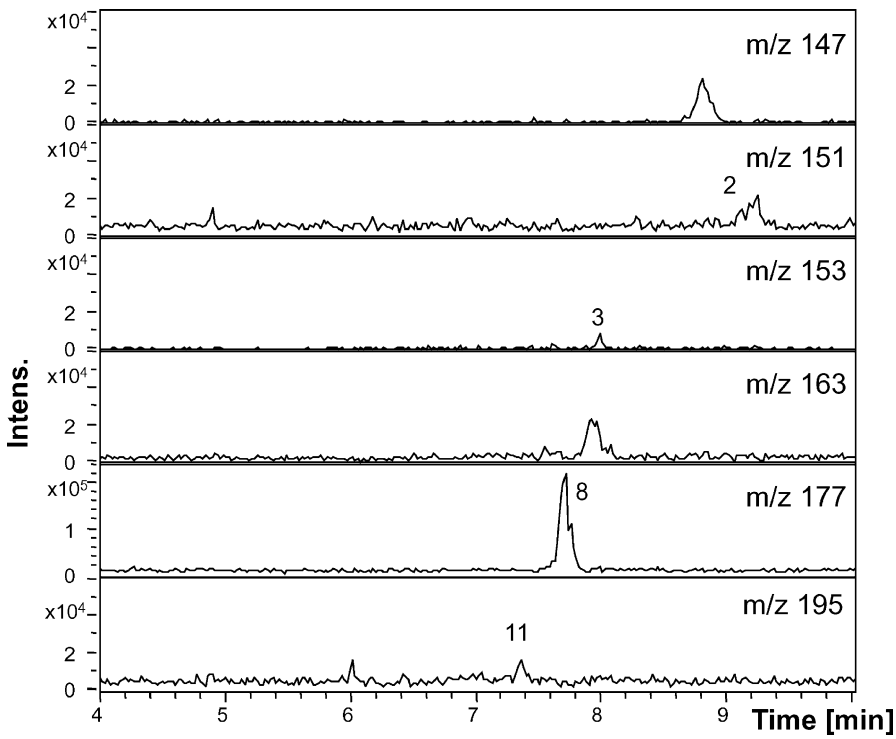

Fig. 6. An example of a biomass burning aerosol sample (pine wood) analyzed by the proposed method. Conditions as in Fig. 2; peak identification as in Table 1.

Table 4
Concentration and RPD (%) of detected compounds (pine wood)

Peak no.	Compound	Ammonium acetate based BGE (μM)[a]	Ammonium hydroxide BGE (μM)[b]	Relative percentage difference (RPD)
2	Vanillin	2.4	2.3	4.3
3	Syringol	Not detected	2.1	Not available
7	3-Hydroxy-4-methoxy benzoic acid	3.7	Not detected	Not available
8	Coniferyl aldehyde	10.8	11.6	7.1
11	3,5-Dimethoxy-4-hydroxyacetophenone	1.8	1.6	11.8

RPD (%) = ABS(100 × ($C_{AA} - C_{AH}$)/(0.5 × ($C_{AA} + C_{AH}$))) where C_{AA} and C_{AH} are the measured concentration of detected compounds in the same sample extract using ammonium acetate and ammonium hydroxide, respectively.

[a] Conditions as in Fig. 1.

[b] Conditions as in Fig. 2.

significant number of unidentified compounds were also found for both methods (data not shown). Attempts to identify unknown compounds in the size segregated biomass burning aerosol from various wood types are in progress.

4. Conclusions

The CZE/ESI-MS methods developed in this study demonstrates a successful determination of substituted methoxy phenols and aromatic acids. A good linearity was found for all analytes in the range of 1–50 μM for ammonium acetate based BGE and 1–40 μM for the ammonium hydroxide BGE. A good detection limit was achieved for all the compounds tested. The proposed methods are simple and offer a good reproducibility. The method can be applied to the analysis of complicated biomass burning aerosol samples without interference from the matrix. A detailed characterization of size segregated biomass burning aerosols from various wood types is in progress. A separate paper concentrating on the particle phase emission rates of inorganic ions, dicarboxylic acids and WOSC from various wood types are in preparation.

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