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TAR SAMPLING BY SOLID PHASE MICRO EXTRACTION IN BIOMASS COMBUSTION

Vanessa Marçal Avelar, vanessa.avelar@gmail.com
Marcelo Risso Errera, errera@ufpr.br
Karina Scurupa Machado, ksmachado@hotmail.com
Ana Flávia Locatelli Godói, afgodoi@ufpr.br
Sandro José Froehner, froehner@ufpr.br
Federal University of Paraná, Environmental Engineering Department, Centro Politécnico, 81531-990 Curitiba, PR, Brazil

George Stanescu, stanescu@demec.ufpr.br
Federal University of Paraná, Mechanical Engineering Department, Centro Politécnico, 81531-990 Curitiba, PR, Brazil

Guenther Carlos Krieger Filho, guenther@usp.br
State University of São Paulo, Mechanical Engineering Department, R. Prof. Mello Moraes 2231, Cidade Universitária
05508-900 - Sao Paulo, SP, Brazil

Abstract. *Tar emissions is still a great challenge in biomass combustion. While most gasses can be directly monitored by gas analyzers, tar compounds still hard to be analyzed in real time due to a broad range of compounds, the sampling and analysis process it takes. In this paper we present an apparatus to sample tar that is simpler than usual methods adopted in Brazil. We made use of the sampling method known as solid phase micro extraction (SPME) to replace the tar measurement standard (TMS) for sampling and analysis (van de Kamp et al., 2005). The apparatus collected compounds in free vapor phase and adsorbed in particulates as well. Compounds were analyzed by GC-FID. Biomass burnt was prepared logs of Mimosa scabrella Bentham, known as bracatinga. The furnace operated with concurrent flow and forced exhaustion with nominal power of 70 kW. The new apparatus has shown to be practical, less costly. more reliable when compared to TMS.*

Keywords: biomass combustion; tar sampling; solid phase micro-extraction; emissions

1. INTRODUCTION

Biomass burning is of great interest since it is renewable and it is often available as process or manufacture waste. A recent development in biomass concurrent furnaces (Klautau *et al.,* 2008) for grain drying plants has shown to be promising with regard to emissions (very low particulates and carbon monoxides) and temperature control (stabilized). Tar levels, however estimated low, were not directley measured.

In fact, one of the lasting challenges of biomass combustion is to eliminate, or reduce to very low levels, tar emissions. Of special interest are applications in which there are direct contact between exhaust gasses and a food product.

In Klautau *et al.*, 2008 tar levels were estimated by an empirical correlation with carbon monoxide (CO) concentrations (Bussmann, 1988) and required further proof even with after Bignal *et al.* (2008) also verified such hypothesis and proposed an alternative correlation.

Tar is considered to be mixture of organic compounds mainly polyciclic aromatic hydrocarbons (PAH) of higher molecular weight. Li and Suzuki (2009) also states the definition or description of tar is not strict since it is usually associated with the fuel (feed) and with the converting process.

The ultimate goal of the monitoring emissions is to determine chemical compounds and their concentrations in the exhaust gasses. One can make use of sensors (known as inline gas analyzers) that provide real-time reading of most common species such as nitrogen oxydes, carbon monoxyde and dioxyde and oxygen. For tars, however, that would be rather expensive, complex and even impossible due to a great variety of compounds tars are made of. Therefore usually tar is firstly sampled, stored, and later analyzed.

One of the methods widely adopted for tar sampling has been the "Tar Measurement Standard" (van den Kamp *et al.*, 2005) that was originally devised for biomass gasification systems and alternatively the one proposed by Simell *et al.* (2000). They are both based on "cold trapping" principle in glass impingers. On the other hand, van der Hoeven (2007) and Brage *et al.* (1997) made use of solid adsorption system.

This paper reports tests of a sampling apparatus and procedure devised to determine tar contents of biomass combustion gas. The system is comprised of a solid phase micro-extraction (SPME) and particulate microfilter, with the conventional ancillary devices. Sampled material is analyzed by Gas Chromatography - Flame Ionization Detector (GC-FID).

The SPME technique is based on the principle of analyte adsorption on a liquid phase coated with a fused silica fiber. According to Brage *et al.*, 1997, this technique was first developed by Berlardi and Pawliszyn, 1989.

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The SPME technique has been also succesfully used to detect traces of organic compounds in other fields of interest (Augusto *et al.*, 2000).

Results were obtained by using a variation of the method by van der Hoeven, 2007 and Brage e*t al.*, 1997. Wood was burnt in small logs in a concurrent (downdraft) furnace as in Klautau *et al.* (2008). Particulates with tars adsorbed were sampled in microfilter while gas phase (condensable) tars were collected by solid phase micro-extraction (SPME).

2. MATERIALS AND METHODS

In this section we describe the methods selected to evaluate tar contents of combustion gas, the sampling apparatus and the operations conditions of the tests.

In short, the method of identifying and quantifying tar compounds consists of sampling tar from the flue stack of a concurrent downdraft woodburning furnace operating at steady-regime. Solid phase and gas phase were sampled to be later analyzed by gas chromatography (GC). Cumulated volumetric dry air flow and condensed water were measured and errors and uncertainties were also estimated.

Operation of the furnace was monitored by a gas analyzer (inorganic compounds) and thermometers in real time thus permiting control of the regime. Three runs were carried out to estimate the optimal sampling time in regard to saturation of the SPME probe. Overall, the method followed van der Hoeven, 2007 and Brage e*t al.*, 1997.

Details of the procedures are presented in section 2.5. We next decribe the experimental apparatus set for this study.

2.1. Concurrent wood burning furnace

The furnace operated during the test was built and validated previously and results were reported in Klautau *et at.*, 2008. It works on induced concurrent flow regime (downdraft) with an adiabatic zone where most of exothermic reactions take place. The furnace was built in house with commercial refractory bricks and plates. Inside walls of the combustion chamber were lined with ceramic fiber paper to function as first insulation layer. Outside walls were covered with ceramic fiber insulation and aluminum foil. Wood logs were manually fed by the top in prescribed regime. The grate area was 0.13 m^2 and the flue stack diameter 0.30 m. The feeding regime was set to a nominal power of the 70 kW.

Thermometers (K- and S-type thermometer) were installed in order ensure full monitoring of the temperature regime. The second control parameter was the carbon monoxide levels that was read every 05 minutes. Temperature at the bottom of the flue stack was controled to be near 800 °C, while CO was held as low as possible. Details and position of the measuring points were reported in (Klautau *et al.*, 2008). Temperature data was collected by a device called Compact Fiel Point (cFP) from National Instruments, and handled by LABVIEW.

The furnace was fed by logs of *Mimosa scabrella* Bentham in pieces of nearly 19 ± 0.8 cm by 3.5 ± 0.6 cm.   The summative and elemental composition of this wood are shown respectively in Tab. 1 and Tab. 2.

Table 1 – Summative analysis of bracatinga

Component	Average Quantity (%)
Celulose – $C_6H_{10}O_5$	51.25
Hemicelulose – $C_5H_8O_4$	21.86
Lignine – $C_9H_{10}(OCH_3)_{0.9-1.7}$	22.76

Source: Romanowski, 1986

Table 2 – Elemental analysis of bracatinga

Component	Average Quantity (%)
C	48.3 ± 1.1
O	43.1 ± 0.12
H	6.04 ± 0.77
Ashes	*0.88 – 1.67

* variable according to edafoclimatic conditions and age. Source: LEB, 2010

Moisture content of the fuel was estimated according to NBR 7190/97, that is, wood log samples were weighted in a analytical scale (BIOPRECISA, model FA2104N, precision of 0.0001 g), then dried in an oven (NOVA ÉTICA modelo 400N) set at 103 °C, then weighted again. Details can be found in Avelar, 2010. Moisture contents of the fuel of each tests will be shown in Section 3.

Feed loads were prepared and sequentially sorted before tests. Feed load was weighted in hook type scale (KERN, model CH15K20, range up to 15 kg e precision of 10g).

Inorganic gas compouds were directley measured by a gas analyzer model TEC-GA12, manufactured by MADUR. The sensors operate under electrochemical principles. Oxygen was read in volumetric percentile with 0.01% precision,

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while carbon monoxide was read in volumetric ppm with 1 ppm precision. Other components were estimated by stoichiometric balance.

We next describe the tar sampling apparatus.

2.2. The tar sampling apparatus

A schematical view of the sampling apparatus is shown in Fig. 1. The piping is copper-made. The tar sampling apparatus is a set of components and devices that starts at the gas collecting probe in the flue stack, followed by a thermocouple, then a filter for solid particles. Next along the piping, comes two collecting points with SPME probes and a thermocouple in between. The apparatus continues with ancillary parts, namely, a condenser and air-moisture separation bottle, then a vacuum pump, a gas flow meter and a thermometer.
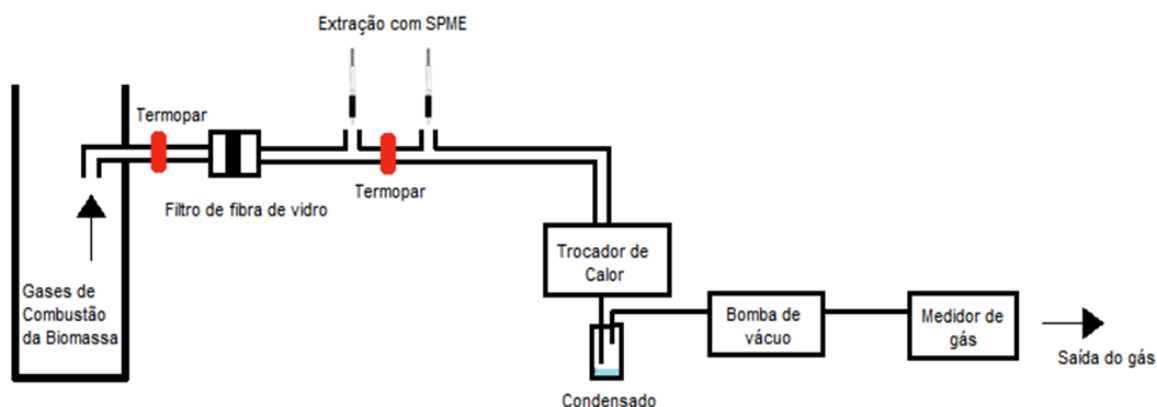

Figure 1. Schematical drawing of the tar sampling line

The particulates filter is a glassfiber micro-filter with 25 mm diameter from Macherey-Nagel®. It sustains up to 500°C.

The SPME device utilizes a polimer coated fiber in order to concentrate volatile and semi-volatile organic compounds in a single step extraction. The fiber was selected to compatible with the substances of interest. The stationary phase used was a melted silica coated with 100μm thick layer of polimeric sorbent, that in this work was polidimethilsiloxano (PDMS), acquired from Supelco.

The SPME probe assembly are shown in Fig. 2. Two probes are inserted simultaneously. Each SPME probe is inserted through a rubber septum in the gas piping. A thermocouple helps make sure temperature is below 280°C. A wet cloth covers the piping in order to hold constant temperature below limits.

A third stage of the sampling line consists of a condenser made of an aluminum coil imersed in a cold pool in order to condense water (to dry up the gas) that is followed by at gas/water separator (bottle). Cummulated water is later weighted and registered.

The sampling line is driven by a vacuum pump that is followed by gas volumetric flowmeter (with 0.00005 m^3 precision) and ultimately a conventional mercury thermometer (precision 0.5 °C) at the gas outlet.

2.3. The Chromatographer

The chromatographer is a Hewlett-Packard 7890 Series GC system coupled to a flame inoinization detector from Agilent Technologies. The colunm is a HP-5 FID (30.0m × 320μm × 0.25μm; 5% molal fraction of phenil-methil-siloxano).

2.4. Methods and procedures

We established a set of procedures for the tests: at set-up phase (1) we check the safety material and conditions, then weigth the feed loads and save three log samples in a plastic bag to the moisture test. The filter element and the condensate reservoir (bottle) are weighted before assembled in the piping. On regard to instruments, we check each one of the thermocouples for circuitry and position in the apparatus and set-up and test the Labview application (VI). We also check for leaks in the sampling piping. The vacuum pump and the gas flowmeter are also inspected. The condenser pool is then filled with water and ice. We condition SPME fibers according the maker's specifications, that is, to inject in CG at 250°C under nitrogen gas flow for 30 minutes in order to remove impurities adsorbed in the fiber

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and free adsorption sites. Two last items are checked, namely: if the particulates filter medium is properly assembled at the support and if the gas analyzer probe is correctly positioned at the flue stack and ready to register data.

Figure 2. Two SPME devices inserted through septum in the gas sampling piping.

The operation phase, (2), starts with powering the exhaust fan then igniting the furnace with alcohol, then Labview application begins monitoring temperatures. Logs are fed freely until the whole furnace reaches the temperature and CO levels of interest, from that point, logs are fed in a prescribed pace to make sure the furnace operates under the desired feeding regime. After that, when fluctuations in temperature and in CO concentrations are within prescribed range, we turn on the vacuum pump, we check pressure reading (gas analyzer) and insert SPME fibers, we read the gas flowmeter and start timing the extraction period. After the prescribed time (60 minutes, determined by the asymptotic time at which tar amounts no longer vary) the fibers are retrieved, the applicator dissambled and the sample is directed straight to the chromatographer laboratory. Time is reseted when a second SPME fiber is inserted in the line and a new reading of the gas flowmeter is done. During the entire period inorganic compounds are periodically read by the gas analyzer.

After samples are collected, there still are few steps to close the test (3): the sampling line is first turned off and the condensate bottle is disconnected and sealed. Feeding is terminated and after and flue stack cools down, the filter element suport is disassembled and the filter element is stored. Finally, all the devices and equipments can be turned off.

Samples can now be analyzed (4): we weight the filter element and the condensate bottle, and the remaining char and ashes as well. Chemical analyses will be described next.

The SPME fibers with tar sampled are directed to chromatography laboratory to undergo analysis of PAHs. The standard solution utilized to identify and quantify chromatographic peaks comprises sixteen PAHs compounds and it was prepared by Accustandard, with 99.9 % of purity.

Chromatographic conditions. PAHs were analyzed with the following operating parameters: the temperature program for operation of the GC-FID was the following: initial temperature set as 45°C and held by two minutes, then temperature raised up to 150°C in a 13°C/min pace and held for one minute. Another temperature raise up to 200°C in a pace of 5°C/min, then held by 5 minutes. A final heating up to 280°C in a 5°C/min pace and holding for 1 minute. The total run time in CG was of 43 minutes for each sample. A constant flow rate of 1ml/min of nitrogen gas past through the column. The sample size was 1 μL. Injection took place in splitless mode.

The procedure to analyze the tar in solid phase trapped it the filter is the following: the micro filter is removed from its support then undergo drying for 2 hours at 40°C in order to remove residual moisture. Afterwards the micro filter element is weighted and its weight is compared with the reading prior to the test run. PAHs adsorbed in the particulate matter collected by the filter element were analyzed following the procedure described by Castellano, 2003.

In order to extract the PAHs from the filter, the filter is "minced" " triturado " then a 20 mL of di-chloromethane of degree HPLC is added. The mixture remained under ultra-sound agitation for a 15 minutes period. The yield extract was then filtered in nithrate membrane with porosity of 0.45 μm for three times. This time the yield are mixed and evaporated until aproximately 1mL. This last extract is then purified in a preparatory chromatographic column with 2 g of active sylica, 2 g of alumin oxide e 1 g of sodium sulphate. The elution of PAHs in the preparatory chromatographic column constituted of 20 mL of a 1:1 di-chloromethane and hexane. The eluate was then evaporated and injected in CG/FID.

The last sample processed was the condensate retained at the bottle. It was weighted before the sampling test and then weighted again in the analytical scale. The amount of condensate was also used to bound estimates by

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stoichiometric relations since it was compared with the overall moisture (wood and air) intake in the furnace and the water produced in the combustion processes.

Errors of tar concentrations and amounts were considered and uncertainties were estimated by known methods of uncertainty calculation.

Results were summarized in tables and charts and compared to previous studies that used same fuel in furnaces of similar power.

3. RESULTS AND DISCUSSION

In this section we present the results of the full analysis of the exhaust gasses of burning *bracatinga* in concurrent wood burning furnace. For the sake of consiceness, only results of run (3) will be shown.

We first present operational indicators such temperature and non-organic composition of combustion gasses in Table 3 and the temperature at the transition of the the combustion chamber and the flue stack and the carbon monoxide variations during the test #03 in Fig. 3. That shows combustion conditions were under steady regime.

The water balance in the test runs was shown in Table 4. It was used to bound measurements and calculations. The sixth column shows the calculated gas volume that past through the flue stack, while the fifth column only the gas past the sampling piping.

Two chromatographic charts are shown if Fig. 4, namely, (a) gaseous phase PAHs peaks of SPME sample and (b) PAHs peaks for the solid-phase (particulate) sample, (c) n-alkanes peaks for the solid-phase (particulate) sample.

The total amount of PAHs in the gas phase (sampled by SPME probe) was of 0.4127 µg/Nm^3. Bignal *et al.* (2008) operated a 50kW water boiler in approximately levels of carbon monoxide and temperature with three different types of biomass and sampled tar by impinging bottles. In the corresponding test (@96 ppmv of CO), they obtained 0.2 µg/Nm^3 of total gaseous phase tar. Our estimatives are in agreement within a acceptable range given the differences in the furnace, the fuel and the operation.

We also compared our results to another work (Atkins *et al.*, 2010) that used the same tecnique as Bignal *et al.*, 2010 to sample tar while burning pellets. When PAHs compounds are classified with respect to the number of rings (nuclei), one can present the results as shown in Fig. 5. While Atkins *et al.*, 2010 point out that the major component of tar is naphtalen (2 aromatic rings), in all three test runs, the most abundant PAH compound found in our test was fenanthreno (3 aromatic rings). Besides naphtalen, our results can be considered close enough.

The particulate phase analysis resulted in 0.0083g of sampled material. PAHs and n-alkanes were extracted as presented in section 2. The concentration of total PAHs in the solid phase was of 1.57 µg/Nm^3, while in Bignal *et al.,* 2008 it was 1.2 µg/Nm^3. We also sorted the PAHs by the number of the rings and compared to the literature. In this case we one can only find agreement in magnitude sense.

We finally computed the total tar (gaseous and solid phase): 1.98 µg/Nm^3 while Bignal *et al.*, 2010 found 1.4 µg/Nm^3 at the corresponding level.

4. CONCLUSIONS

In this paper we tested an alternative method to sample gaseous phase tar by a solid adsorption micro-extraction (SPME) probe in the associated procedures. The sampling device was tested in a concurrent wood burning furnace fed by "bracatinga".

We found it to be low cost (SPME costs near US$ 250.00 and are not disposable) and feasible to adapt once a chromatograph laboratory is readily available.

On the comustion side, burning wood logs (slumber) of bracating in our concurrent furnace performed well in temperature control while carbon monoxide and tar levels were low. As matter of fact, emissions were comparable with pellets burning in boiler (Bignal *et al*., 2008 and Atkin *et al.*, 2010).

In sum this paper shed light in the SPME sampling procedure and the performance of bracatinga wood logs as a potential fuel.

5. ACKNOWLEDGEMENTS

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Table 3 – Operating indicators of the test run #03

Test run #03	$\dot{m}_{wood}$ = 0.3 kg/min
T_{amb}(°C)	22
$T_{flue\ stack\ base}$ (°C)	804(σ=37)
$T_{flowmeter\ outlet}$ (°C)	27
Air humidity (%)	53
Ambient Pressure (hPa)	1021
Total of Wood logs used in regime (kg)	19.5
Time period under steady regime (min)	62
Total particulate matter (g/Nm^3)	0.0059
Condensate (g/Nm^3)	58.93
Carbon (%)	49.1
Hydrogen (%)	6.4
Oxygen (%)	43.2
Ashes (%)	1.3
Humidity dry base (Ws) - (%)	20.2
Humidity wet base (Wu) - (%)	16.8
HHV (MJ/kg)	15.96
LHV (MJ/kg)	11.70
Nominal Power (kW)	58.52
Total mass of combustion gasses (kg$_{flue}$/kg$_{fuel}$)	13.13
N_2 (%) - (kgN$_2$/kg)	66.88 – 8.78
H_2O (%) - (kgH$_2$O/kg$_{flue}$)	5.72 – 0.75
O_2 (%) - (kgO$_2$/kg$_{flue}$)	11.02 – 1.44
CO_2 (%) - (kgCO$_2$/kg$_{flue}$)	16.36 – 2.14
CO (%) - (kgCO/kg$_{flue}$)	0.00113 – 0.00014
combustion gasses (kg/s)	0.0656
Air excess	1.93
Enthalpy of combustion gasses (kJ/kg)	1049.93
Mean concentration CO (ppmv)	119(σ = 20)
Mean concentration O_2 (%)	10.1(σ = 1.61)

Table 4 – Relation between the condensed water vapor retained and measured

Run	Retained Condensate (g)	Wood (kg)	W_u (%)	Vol. Gas (meas.) (Nm^3)	Vol. Gas (calc.) (Nm^3)	Steam (meas.) (g/Nm^3)	Steam (calc.) (g/Nm^3)
03	58.9388	19.5	16.8	2.931	311.72	20.10	27.17

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(a)

(b)
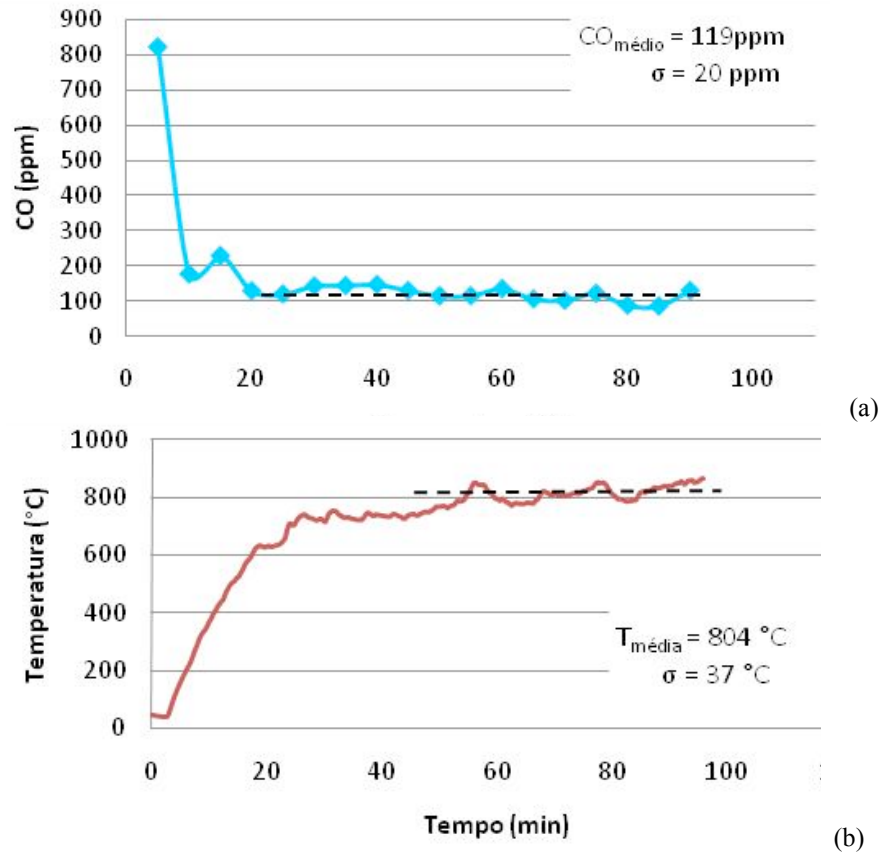
Figure 3. (a) Levels of carbon monoxide during test; (b) Temperature variation during test.

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Sinal frontal Results

Pk #	Name	Retention Time	Area	Concentration
2	NAFTALENO	7.715	2077	0.000
4	ACENAFTALENO	10.567	632	0.000
6	ACENAFTENO	10.735	3730	0.000
14	FLUORENO	12.054	15739	0.000
28	FENANTRENO	14.775	5252	285.616
30	ANTRACENO	15.284	2184	160.123
32	FLUORANTENO	19.834	52	0.000
35	PIRENO	20.280	0	0.000
67	BENZO- A ANTRACENO	27.674	917	66.991
70	CRISENO	27.993	2003	0.000
82	BENZO-B FLUORANTENO	30.980	5087	0.000
90	BENZO-K FLUORANTENO	34.325	43	0.000
96	BENZO-A PIRENO	35.062	7	0.000
110	INDENO	39.708	3261	0.000
111	DIBENZO-A,H ANTRACENO	40.126	3118	3.578
113	BENZO-GHI PERILENO	40.618	42200	263.008

(a)

Sinal frontal Results

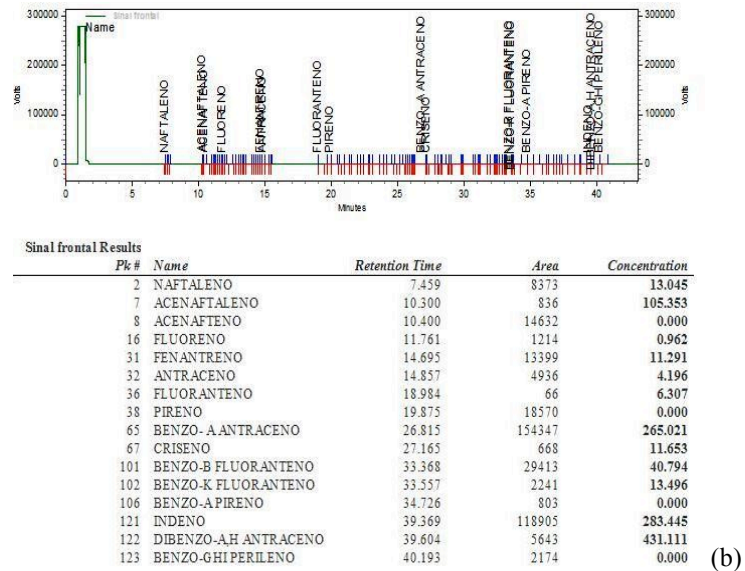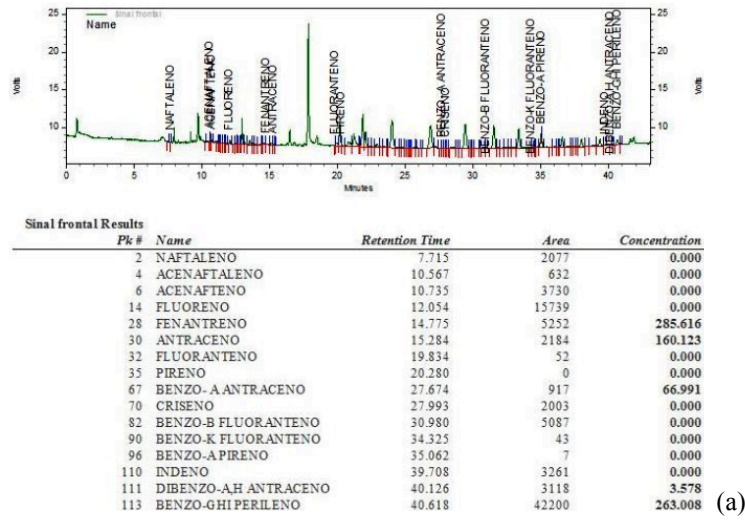| Pk #	Name	Retention Time	Area	Concentration
2	NAFTALENO	7.459	8373	13.045
7	ACENAFTALENO	10.300	836	105.353
8	ACENAFTENO	10.400	14632	0.000
16	FLUORENO	11.761	1214	0.962
31	FENANTRENO	14.695	13399	11.291
32	ANTRACENO	14.857	4936	4.196
36	FLUORANTENO	18.984	66	6.307
38	PIRENO	19.875	18570	0.000
65	BENZO- A ANTRACENO	26.815	154347	265.021
67	CRISENO	27.165	668	11.653
101	BENZO-B FLUORANTENO	33.368	29413	40.794
102	BENZO-K FLUORANTENO	33.557	2241	13.496
106	BENZO-A PIRENO	34.726	803	0.000
121	INDENO	39.369	118905	283.445
122	DIBENZO-A,H ANTRACENO	39.604	5643	431.111
123	BENZO-GHI PERILENO	40.193	2174	0.000

(b)

Figure 4. Chromatographic peaks, retention times, peak area and resulting concentration of
(a) each PAHs of SPME sample; (b) each PAHs of Extraction of particulate matter;

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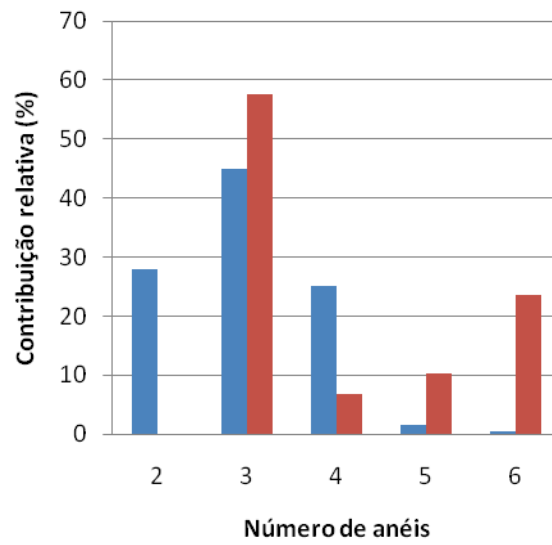October 24-28. 2011. Natal. RN. Brazil

Figure 5. Comparison of total tar in gaseous phase sorted by the number of aromatic rings:
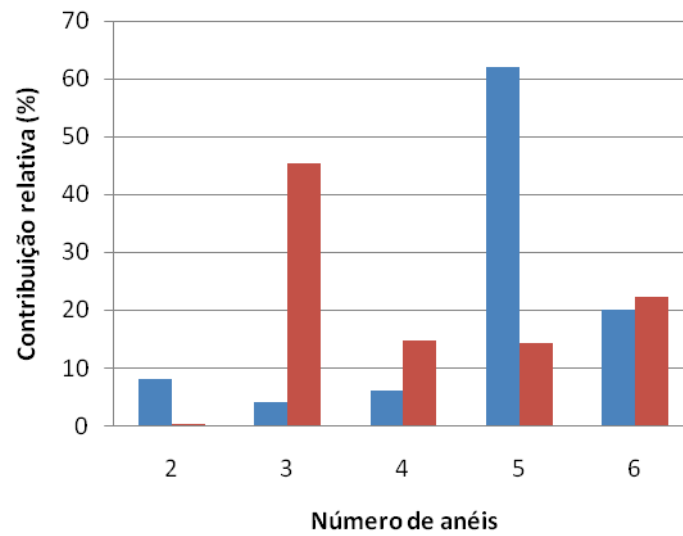(red) the present work; (blue) Atkins *et al.*, 2010

Figure 6. Comparison of total tar in particulate phase sorted by the number of aromatic rings:
(red) the present work; (blue) Bignal *et al.*, 2010