Obtaining polycyclic aromatic hydrocarbon concentration ratios and molecular markers for residential wood combustion: Temuco, a case study

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TECHNICAL PAPER

Obtaining polycyclic aromatic hydrocarbon concentration ratios and molecular markers for residential wood combustion: Temuco, a case study

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It is known that residential wood combustion (RWC) is an important source of fine particle emissions. The purpose of this work was to characterize the chemical composition of the particulate matter present in the Temuco urban atmosphere during winter, specifically the polycyclic aromatic hydrocarbon (PAH) profile, because PAHs are considered to be among the key compounds in particulate matter toxicity. During the 2008 winter monitoring campaign, samples of particulate matter with aerodynamic diameters of ≤10 (PM_{10}) and ≤2.5 ($PM_{2.5}$) µm were taken on days with contamination episodes. Sixteen U.S. Environmental Protection Agency (EPA) PAH compounds were extracted with toluene and determined by gas chromatography–mass spectrometry (GC-MS). The results show that phenantrene was the predominant compound associated with particulate matter, at a concentration range between 300 and 600 ng m^{-3}, 18 times higher than the second most abundant PAH compound. High-molecular-mass compounds such as dibenz[a,h]anthracene, benzo[g,h,i]perylene, and indeno[1,2,3,c,d]pyrene were also found, but they were minorities in the set. It was recognized from the PAH concentration ratios of the Temuco atmospheric aerosol that the main contamination source was in fact residential wood combustion; although not all the concentration ratios evaluated match the reported reference values, probably due to the kind of biomass used, the characteristics of Chilean heating appliances and climate.

Implications: The study results suggest that if molecular markers (MMs) and concentration ratios (CRs) are dependent on the type of firewood and combustion processes specific to each location, among others factors, then the values of these indicators could be applicable with caution between different areas, although not necessarily they must be totally in accord. Moreover, having representative values of MM and CR of each place, it will achieve a better estimation of the source importance and concentrate the monitoring and the control strategies. Regulatory organizations in each country can have more reliable information and optimize financial and human resources, enabling it to reduce air pollution and improve the health of population.

Introduction

Among the organic compounds associated with particulate matter (PM), the fraction that was investigated intensively corresponded to polycyclic aromatic hydrocarbons (PAHs), and especially the 16 priority PAHs defined by the U.S. Environment Protection Agency (EPA) (Furuuchi et al., 2007).

The analysis of PAHs in the atmosphere is of great concern due to their adverse health effects. The carcinogenicity and mutagenicity of these compounds are recognized, with benzo[a]pyrene (BAP) being the most widely studied; in fact the European Union (EU) has set limit values for the total BAP content in the PM_{10} fraction in ambient air averaged over a calendar year at 1 ng m^{-3}] (Machala et al., 2001; Pufulete et al., 2004). PAHs are semi- to low-volatile and lipophilic organic pollutants generated by hydrolysis and incomplete combustion of organic material, and the carbonization process (Shi et al., 2009). Although they arise from natural sources, such as forest fires and volcanic eruptions, their principal sources are anthropogenic emissions such as the combustion of fossil fuels (diesel and gasoline) in mobile and stationary sources, and biomass (wood). PAHs, in the form of both gases and particulate matter, may be discharged into the atmosphere and transported over long distances, resulting in widespread distribution on a continental scale.

The ratio values of individual PAH species and molecular markers in ambient samples are frequently employed as

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diagnosis tools to identify the impact of different sources of PAHs in ambient air (Fang et al., 2004; Kavouras et al., 2001; Martis et al., 2005). However, both concentration ratios (CRs) and profiles should be used with caution, for several reasons: the reference CR values were normally obtained for fuels and processes in the northern hemisphere, and are not directly transferable to the situation in other parts of the world such as South America; CRs can be altered by mixing phenomena and the reactivity of some PAH species; the reference intervals for CRs overlap (Zou et al., 2003) In combustion processes, the relative percentages of PAHs depend on the chemical composition and combustion conditions of the fuel, so in order to diminish errors in the estimates, some researchers recommend the use of supplementary markers, trace elements, or statistical tools (Callen et al., 2010).

Our report was based on Temuco, a Chilean city of more than 260,000 inhabitants with diverse ethnic population, located 667 km south of the capital, Santiago, and 80 km from the Pacific Ocean. Although the economy of the city is mainly based on agroindustry, forestry, and service activities, it is considered to be the Chilean city with the worst particulate matter pollution (Temuco Municipality, 2010). The forests in the Temuco area contain a variety of tree species, such as Roble (*Nothofagus oblique*), lingue (*Persea lingue*), among others. Roble and *Eucalyptus* spp. are the species most commonly used for RWC, especially in fall–winter. Wood combustion is the emission source of a variety of different air pollutants such as fine particles, volatile organic compounds (VOCs), PAHs (estimated contribution 80–95%), among other (Hellen et al., 2008). According to the latest PM_{10} emission inventory carried out in Temuco, RWC is the principal source of this pollutant, responsible for 93% of emissions. For that reason, this city is considered as a "monosource" case study (Diaz-Robles et al., 2008). Currently the negative effects of exposure to polluted environments on people's health are undisputed, especially those due to inhalable particulate matter ($PM < 10\ \mu m$; PM_{10}). Although few epidemiological studies have been conducted in Temuco to establish the link between air quality and health, a recent study found a strong relationship between PM_{10} and daily mortality cases between 1997 and 2002 among subjects over 65 years old (Sanhueza et al., 2005).

PAHs have been considered as potential wood smoke tracers, although they are not unique to wood combustion. Benzo[a] pyrene and fluoranthene have been reported as tracers for wood combustion (Wang et al., 2007). Other authors found that fluorene, phenanthrene, anthracene, fluoranthene, and pyrene contribute more than 70% of the mass of PAHs emitted by the birch wood combustion (Hedberg et al., 2002). The emissions from softwood and hardwood have been compared, and show different PAH profiles: (a) retene, benzo[g,h,i]perylene, benzo[k+b] flouranthene, benzo[e]pyrene, benzo[a]pyrene, and indeno [1,2,3,c,d]pyrene predominate in softwood combustion, whereas (b) fluoranthene, pyrene, benz[a]anthracene, and chrysene were found during hardwood combustion. Retene, a semivolatile PAH, has been reported from the combustion of conifer trees (Bari et al., 2009). Emissions of PAHs from wood combustion depend on many factors: the type, quality, and moisture content of the wood, the structure and design of the stoves, the air supply,

among other. In view of the complexity of the emissions and the lack of information, the objective of this study was to obtain the characteristics PAH concentration ratios and molecular markers for residential wood combustion in Temuco.

Experimental Methods

Sampling

Four samples of the atmospheric aerosol were collected in a residential zone in Temuco. The city is located 114 m above sea level, in a forested region and set between the Ñielol and Conun Huenu hills, and the Andes Mountain Range. The Las Encinas sampling station, Figure 1 ($38°44'S$; $72°37'W$), is situated in the middle of a vacant lot in a very representative and typical residential area of the city responsible for most of the wood burning, located about 2.7 km west of the city center. The sampling system is located at the same site as the air quality monitoring station belonging to the Health Ministry (Diaz-Robles et al., 2008). The sampling period began on August 11 and lasted up to September 12, 2008. Samples were taken over 24 hours. Using the prognosis model operated by Comisión Nacional del Medio Ambiente (CONAMA) Araucanía, it was possible to take these samples on winter days when PM_{10} levels exceeded the maximum permitted by the National Air Quality Standard ($150\ \mu g\ m^{-3}$, 24-hr average). The fine particulate matter standard will be applied from 2012 ($50\ \mu g\ m^{-3}$ of PM $\leq 2.5\ \mu m$ [$PM_{2.5}$], 24-hr average). Figure 2 shows the hourly profile of PM_{10} and $PM_{2.5}$ on sampling days.

The samples were collected with a new low-volume sampler Partisol 2300 Speciation Sampler (Thermo Electronic Corp., Albany, NY, USA), using a flow of $10\ L\ min^{-1}$ (similar air volume level is breathed by a person West, 2004). The sampler cartridges are equipped with $PM_{2.5}$ impactors, Teflon membrane filters of $0.45\ \mu m$ pore size (TF; Sartorius, Goettingen, Germany) and three polyurethane foam filters (PUFs; 30 mm [height] × 55 mm [outer diameter]; Gore-Tex, Newark, DE, USA), which allows the identification and quantification of PAHs present in the gaseous phase as well as that associated with particulate matter $PM_{2.5}$.

Analysis procedure

A certified standard solution of 16 EPA PAHs was acquired from Dr. Ehrenstorfer, Germany, (acenaphthylene [ACY], acenaphthene [ACE], fluorene [FLU], phenanthrene [PHE], anthracene [ANT], retene [RET], benz[a]anthracene [BAA], chrysene [CRY], pyrene [PYR], methylpyrene [M-Pyr], fluoranthene [FLT], benzo[a]pyrene [BAP], dibenz[a,h]anthracene [DBA], inden[1,2,3,c,d]pyrene [INP], Indeno[1,2,3-c,d]fluoranthene [INFL], and benzo[g,h,i]perylene [BGP]).

The standard reference material "Urban-Dust, SRM 1649," containing PAHs absorbed on particulate matter with a total quantity of PAHs between 0.02 and 0.22 μg, was obtained from U.S. National Institute of Standards and Technology (NIST).

Toluene was supplied by Fisher Scientific (Waltham, MA, USA) and the internal standards 1-methylpyrene, 2,2'-binaphthyl and indeno[1,2,3-c,d]fluoranthene were obtained from Dr. W. Schmidt, Institut für PAH-Forschung, Greifenberg, Germany.
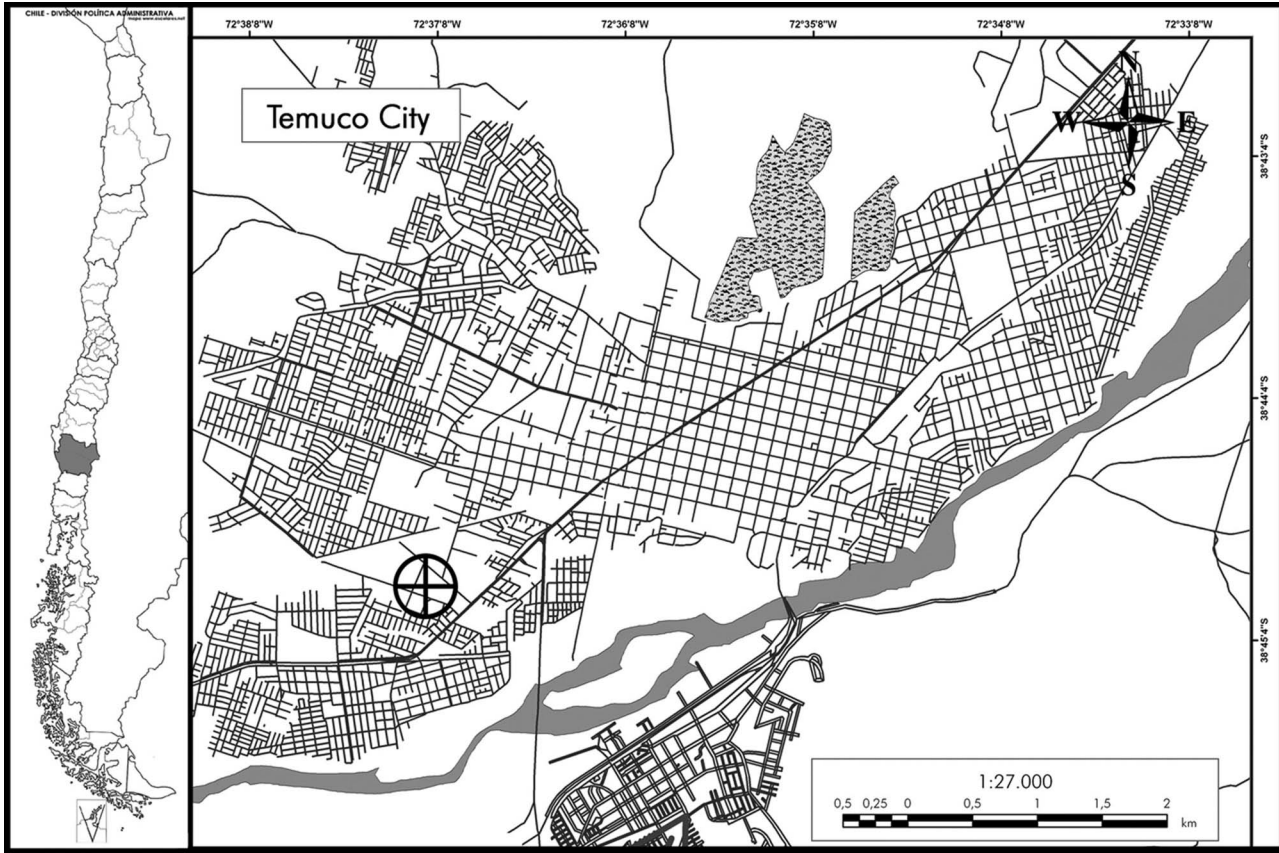
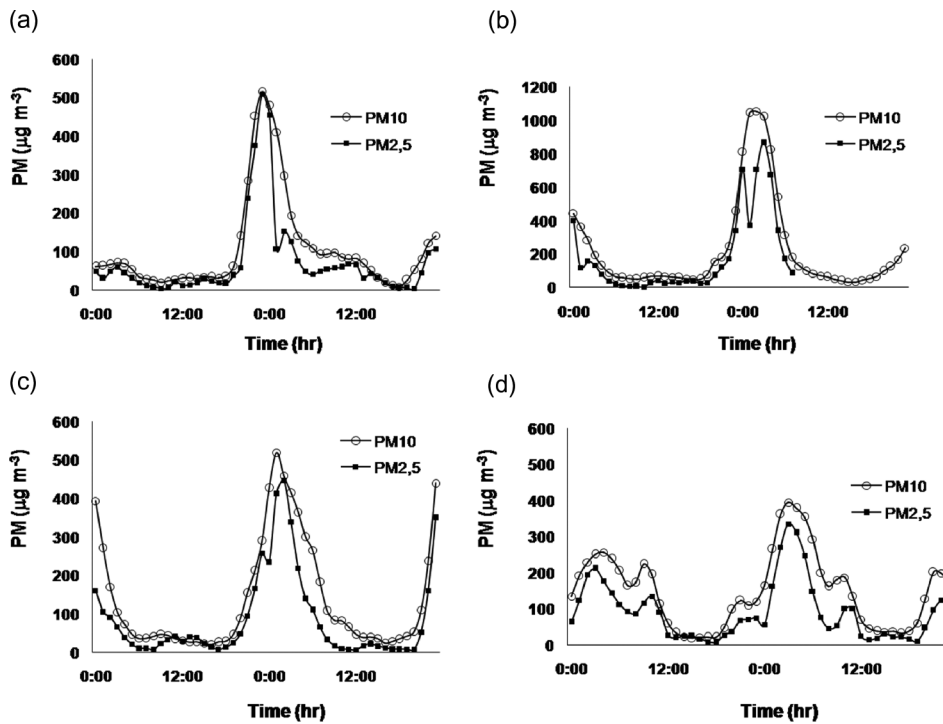**Figure 1.** Map of the location and surrounding areas of the Las Encinas sampling Station (cross).

Figure 2. Hourly profile concentration of PM_{10} and $PM_{2.5}$ on sampling days of the winter monitoring campaign, 2008.

Extraction was done separately for the TF and PUFs. To ensure the quality of the results, each step of the analysis procedure was optimized and validated independently: extraction with toluene of the TF and PUF, volume reduction using a rotary evaporator, evaporation with controlled nitrogen stream, sample clean-up, and chromatographic determination (Cereceda-Balic et al., 2002; Schiappacasse et al., 2006).

The concentration of PAHs in the atmospheric aerosol was determined by a gas chromatographer with flame ionization detector (GC-FID) (Autosystem XL; Perkin Elmer, Arlington, MA, USA); and for structural confirmation, gas chromatographer with mass spectrometric detectors (GC-MS) (Clarus 500; Perkin Elmer) was used. Both instruments were equipped with a programmable large-volume injection technique for capillary gas chromatography with solvent diverts and split/splitless injector with electronic pneumatic control and temperature programming (PSSTM; Perkin Elmer) (Cereceda-Balic et al., 2002; Schiappacasse et al., 2006). A new PSS injector is used on GC-FID to eliminate most of the solvent (toluene) away from the injector (only 6 sec) through the PreVentTM system (Perkin Elmer), so that the analytical column and the detector are able to produce appropriate chromatographic conditions and the peaks in the chromatograms are much more suitable for quantitative analysis. Splitless mode was used to inject 1 μL of the extract into a 30-m-long capillary column of 5% phenylmethylsilicone fused silica (Varian Factor Four type DB-5; Walnut Creek, CA, U.S.A.); temperature program: 60 °C, 3 min, 10 °C min^{-1} → 210 °C, 5 min, 30 °C min^{-1} → 285 °C, 7 min, 15 °C min^{-1} → 300 °C, 10 min; carrier gas: helium for both chromatographic systems. The samples were analyzed by electron impact (EI) GC-MS in scan mode (50–500 Da). The internal standard method was used for PAH quantification, using 1-methylpyrene, 2,2'-binaphthyl and indeno[1,2,3,c,d]fluoranthene (Dr. W. Schmidt, Institut für PAH-Forschung). The limit of detection (LOD = 3SDs) varied between 0.012 (indeno[1,2,3,c,d]pyrene) and 0.094 (chrysene) ng m^{-3}.

Table 1 shows the recovery percentage yield and the detection limit for each standard obtained through the analytical technique used in the study.

Results and Discussion

Characterization of the PAH profile

The PM$_{10}$ and PM$_{2.5}$ daily average concentration for the monitoring period were 153.7 ± 25.6 and 100.8 ± 21.0 μg m^{-3}, reaching hourly peaks values of 1.053 and 865 μg m^{-3}, respectively (Figure 2).

PAH average concentration profile associated to the particle matter PM$_{2.5}$ is shown in Figure 3.

Great concentration variability was observed in the pattern (Table 2), but it is known that when PAHs enter the atmosphere, they undergo processes of transport, dispersion, and ad/absorption on particles and surfaces. For that reason, the distribution of atmospheric PAHs between particulate and gas phases is influenced by several factors, such as PAH vapor pressures (function of the temperature), amount of fine particles (surface area for PAH adsorption), ambient temperature, PAH concentration, and affinity of each PAH with the particles of the organic matrix (Baker and Eisenreich, 1990). Reports regarding the seasonal effect on gas–particle equilibrium variations have been found for the winter months, showing a greater concentration of PAHs in the solid phase (Tsapakis and Stephanou, 2005). Our work was focused only on the winter season, when Temuco faces episodes of high contamination and a greater concentration of PM$_{10}$ and PM$_{2.5}$. For this period, a greater contribution to the total PAH concentration was found from the gas phase (Figure 3) than the solid phase, with the exception of FLT, with a similar contribution of PAHs from both phases (gas and particulate); and BAP and BGP, which had a slightly greater PAH contribution from the particulate phase PM$_{2.5}$, although it is recognized that the sample

Table 1. PAHs recovery yield for two concentration levels and method detection limits (LOD)

PAHs	Abbrev.	LOD (ng m^{-3})	Recovery (%)	
			Level 8 (ng μL^{-1})	Level 80 (ng μL^{-1})
Acenaphthylene	ACY	0.065	91.61	86.45
Acenaphthene	ACE	0.038	111.60	56.23
Fluorene	FLU	0.087	110.17	61.75
Phenanthrene	PHE	0.053	115.53	66.72
Anthracene	ANT	0.061	71.03	68.92
Fluoranthene	FLT	0.035	126.44	86.40
Pyrene	PYR	0.038	73.70	83.30
Retene	RET	0.030	83.57	85.60
Methylpyrene	M-Pyr	0.011	80.72	75.30
Benz[a]anthracene	BAA	0.057	88.84	96.34
Chrysene	CRY	0.094	109.52	99.79
2,2'-Binaphthyl	BIN	0.016	80.75	95.36
Benzo[a]pyrene	BAP	0.051	87.63	98.75
Indeno[1,2,3-c,d]Fluoranthene	INFL	0.099	78.46	94.56
Dibenz[a,h]anthracene	DBA	0.015	79.27	92.81
Benzo[g,h,i]perylene	BGP	0.017	75.63	67.60
Indeno[1,2,3,c,d]pyrene	INP	0.012	76.65	92.41

Table 2. Average period concentrations of total PAHs (TPAHs) and their limits of confidence (95%) for Temuco ($n = 4$), with the PAH concentration distribution between gas-phase (PUF) and particulate matter (TF) PM$_{2.5}$ (TF)

PAHs	PUF (PAHs) (ng m^{-3})	TF (PAHs) (ng m^{-3})	TPAHs (ng m^{-3})	(%)
2–3 rings				88
ACY	17.01 ± 4.14	9.47 ± 5.47	26.49 ± 7.31	4.3
ACE	10.98 ± 8.12	8.50 ± 6.78	19.48 ± 14.41	3.2
FLU	7.66 ± 9.56	5.36 ± 5.31	13.02 ± 7.02	2.1
PHE	373.79 ± 212.90	81.67 ± 78.64	455.45 ± 247.98	74.2
ANT	9.35 ± 7.02	4.90 ± 2.41	14.25 ± 5.33	2.3
RET	7.44 ± 8.59	3.93 ± 3.30	11.37 ± 7.41	1.9
4 rings				9
FLT	9.52 ± 9.44	9.57 ± 6.70	19.09 ± 13.11	3.1
PYR	12.74 ± 16.37	5.29 ± 4.62	18.03 ± 14.13	2.9
BAA	7.46 ± 11.47	2.36 ± 2.03	9.82 ± 11.32	1.6
CRY	4.61 ± 5.35	3.70 ± 3.16	8.32 ± 5.83	1.4
>5 rings				3
BAP	4.86 ± 10.23	5.92 ± 3.81	10.78 ± 10.20	1.8
DBA	3.02 ± 3.60	1.42 ± 0.65	4.43 ± 3.90	0.7
BGP	1.53 ± 0.96	1.57 ± 1.95	3.10 ± 2.20	0.5
INP			n.d.	
$\sum$PAHs	462.32 ± 188.79	148.43 ± 100.31	613.64 ± 258.58	100
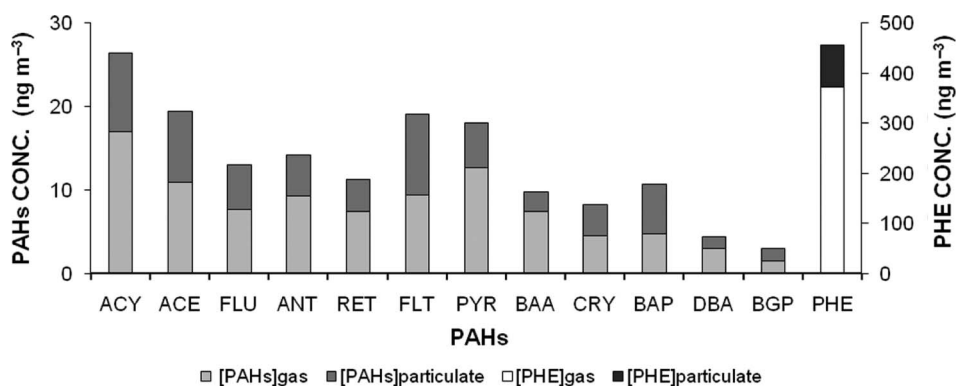
Figure 3. Average total and gas-particulate PAH profile for the Temuco atmospheric aerosol, obtained during the winter monitoring campaign 2008. Black = PHE particulate-phase concentration; white = PHE gas-phase concentration; dark grey = particulate-phase PAH concentration; light grey = gas-phase PAH concentration.

collection method could introduce some artifacts in the gas/particle phase distribution (Arey et al., 1989; Pozzoli et al., 2004; Stracquadanio and Trombini et al., 2006).

Some authors suggest a correlation between the total concentration of PAHs in the solid phase and climatic variables such as moisture percentage, wind direction, and temperature (Walgraeve et al., 2010; Wang et al., 2007). They reported an inverse correlation of the total concentration of polycyclic aromatic hydrocarbons ($\sum$PAHs) with relative humidity and weak correlation with temperature (T). Besides, it has been found an inverse correlation of PM$_{10}$ with the relative humidity and significant correlation with temperature, suggesting a rise in the fine particle grow due to agregation with the relative humidity (RH) and as consecuence, a reduction of PM$_{10}$ concentration and the bounded $\sum$PAHs (Walgraeve et al., 2010). During the present monitoring period, the temperature varied from -0.8 to 16.8 °C, and the humidity from 30% to 99%. These circumstances may account for the great variability found in the average PAH concentrations and the gas–particle distribution profile as shown in Table 2.

However, despite the variability, some common characteristics for all the PAH profiles obtained during the Temuco campaign were observed (Table 2). From all the PAHs analyzed, more volatile PAHs (2 and 3 rings) represent 88% of the total analyzed PAHs, in agreement with other literature wood burning emissions report (Zou et al., 2003). In all the Temuco samples, phenantrene (PHE) was the most abundant species, showing the highest concentration, which represents the 74.2% of all the PAHs analyzed, follow by acenaphtylene (4.3%). In the literature, high concentrations of PHE are typically found in emissions from biomass combustion, particularly wood combustion (Rogge et al., 1998) Four-ring PAHs represent 9%, and high-molecular-weight PAHs (>5 rings) only represent 3% of the total PAH concentrations. The concentrations of genotoxic carcinogenic PAHs, such as BAP, DBA, DGP, BAA, and CRY, were
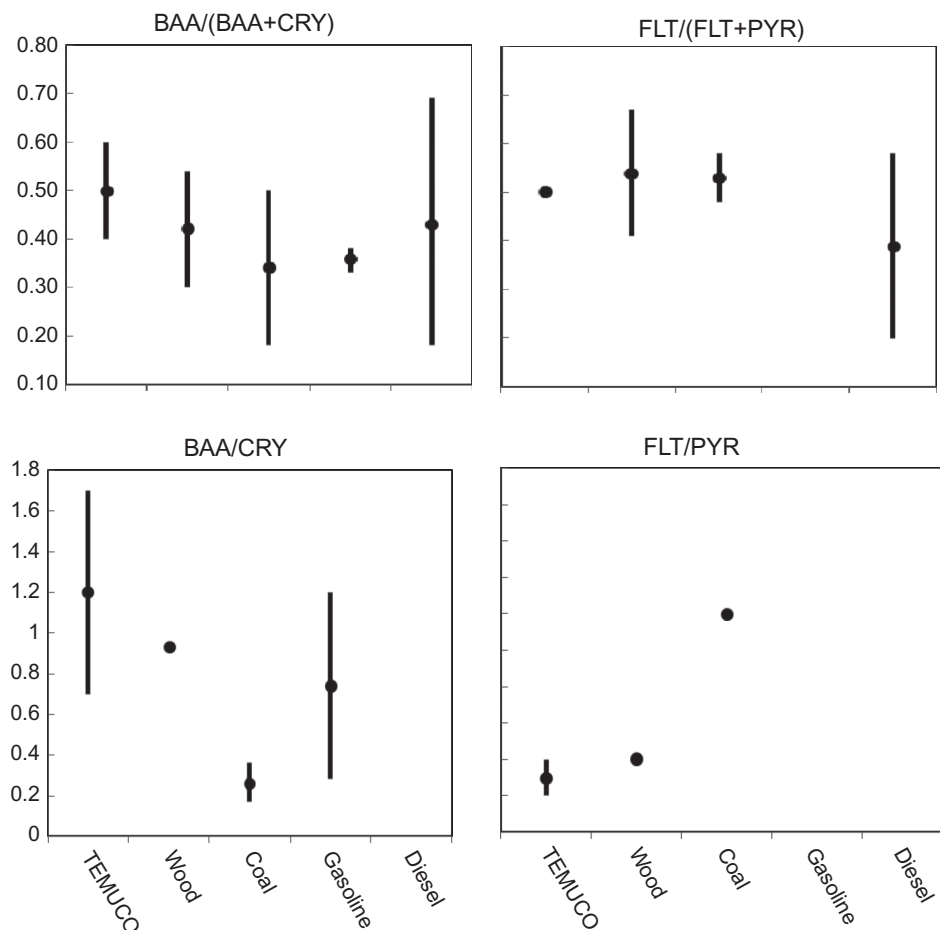
Figure 4. Concentration ratios for TPAHs determined in Temuco 2008, compared with literature values.

identified and the maximum concentration of benzo[a]pyrene (BAP) was 10.78 ng m^{-3}, lower than that reported in a previous study (98 ng m^{-3}) (Schiappacasse et al., 2006). This result could be attributed to differences between the characteristics of monitoring system and monitoring sites in the two studies (Diaz-Robles et al., 2008).

Concentration ratios (CRs)

The concentration ratios calculated in the present study (Figure 4) were compared with the values found in the literature for different emissions sources (Kavouras et al., 2001; Rogge et al., 1993; Rogge et al., 1998); in the case where one emission source is the dominant contributor in an ambient air sample, its PAH profile is probably quite similar to those in the literature.

Various authors have reported the BAA/(BAA + CRY) and FLT/(FLT + PYR) ratios as the principal indicators of wood combustion, and the FLT/PYR and BAA/CRY ratios as complementary indicators (Kavouras et al., 2001; Rogge et al., 1993; wang et al., 2007). These reference values were used in this study; however, it must be borne in mind that they were calculated for the northern hemisphere and that the situation could differ in the southern hemisphere. Figure 4 shows that for Temuco the BAA/(BAA + CRY) ratio is in the range 0.4–0.6 (95% confidence), which contains the literature value for wood combustion (0.56)

(Kavouras et al., 2001; Rogge et al., 1993) and the reference range (0.3–0.54) (Yunker et al., 2002). This implies that concordance does exist between the local situation and the northern hemisphere. The FLT/(FLT + PYR) ratio for Temuco was 0.5 (95% confidence), which is contained in the reference range 0.41–0.67 (Yunker et al., 2002). On the other hand, the same concentration ratio for Temuco (0.5) does not match the reference value (0.74) according to other authors (Simoneit, 1996), indicating that it is necessary to adjust these CRs to Chilean residential wood combustion conditions. The FLT/PYR calculated ratio is a value range of 0.9–1.0, showing again that wood combustion is predominant, since the value reported for wood combustion is 1.0 (Wang et al., 2007). The BAA/CRY reference value (Wang et al., 2007). for wood combustion is 0.93 and for Temuco we found a range of 0.7–1.7, again containing the value reported in the literature and reconfirming that wood combustion is the principal source of PAH emissions in Temuco.

Conclusions

From the monitoring campaign carried out in Temuco during winter 2008, it was possible to characterize the PAH profile generated by residential wood combustion: (1) PHE concentration ranged between 300 and 600 ng m^{-3}, and was, on average,

18 times higher than the second most abundant compound. Concentrations of high-molecular-mass compounds (DBA, BGP, and INP) are minor in the set.

BAA/(BAA + CRY), FLT/(FLT + PYR), FLT/PYR, and BAA/CRY concentration ratios were evaluated for residential wood combustion (from the PAH determination of the atmospheric aerosol of Temuco). The reference values were contained in the values range obtained in Temuco. From the Temuco results, it was possible to recognize that the principal contamination source is the residential wood combustion.

Acknowledgments

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