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RESEARCH ARTICLE

Using native plants to evaluate urban metal pollution and appoint emission sources in the Brazilian Steel Valley region

Guilherme Carvalho Andrade[1] · **Brenda Vila Nova Santana**[1] · **Mirian Cilene Spasiani Rinaldi**[2] · **Sukarno Olavo Ferreira**[3] · **Renê Chagas da Silva**[3] · **Luzimar Campos da Silva**[1]

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Abstract

In southeastern Brazil, the city of Ipatinga is inserted in the Steel Valley Metropolitan Region, which hosts the largest industrial complex for flat-steel production in Latin America, while also having one of the largest vehicle fleets in the entire country. Since potentially toxic elements (PTEs) are not emitted solely by industries, yet also by vehicular activity, the predominant emission source can be determined by evaluating the ratio between different elements, which are called technogenic tracers. We performed a biomonitoring assay using two tropical legumes, *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya*, aiming to assess chemical markers for the origin of emissions in the region, distinguishing between different anthropogenic sources. Plants were exposed for 90 days in four urban sites and in a neighboring park which served as reference. After the experimental period, plants were evaluated for trace-metal accumulation. *L. ferrea* var. *leiostachya* retained lower amounts of metals associated with vehicular and industrial emission. The opposite was found with *P. echinata*, a species which should be recommended for biomonitoring of air pollution as a bioaccumulator. Plants of *P. echinata* were enriched with Fe, Al, Ni, Cr, and Ba, whereas plants of *L. ferrea* var. *leiostachya* were enriched with Fe, Cu, and Co. In both species, Fe was the element with which plants were enriched the most. Plants showed highest iron enrichment at Bom Retiro, the site downwind to the steel industry, which has shown to be the main particle emission source in the region.

Keywords Biomonitoring · Potentially toxic elements · Iron · Source apportionment · Technogenic tracers · Trace metals

Introduction

Particulate matter (PM) is one of the major byproducts of industrial and vehicular activity. Approximately 1% of PM is formed by metals (USEPA 1996). In addition, PM, especially PM2.5, may carry potentially toxic elements (PTEs) adsorbed to its surface (Liu et al. 2018a; Ali et al. 2019), in amounts that may reach up to 30–35 µg m−3 (Schroeder et al. 1987). Fine particles with associated PTEs may lead to the development of cancer and respiratory and cardiovascular diseases, among other infirmities in humans (Kampa and Castanas 2008; Chen and Lippmann 2009; Cakmak et al. 2014; Ali et al. 2019; Karzai et al. 2021).

PTEs are emitted not only by industries but also by vehicular activity (Kassomenos et al. 2014; May et al. 2014; Kończak et al. 2021). As a result, PTE presence in urban atmosphere may owe to a mixture of compounds originated from those two sources. The predominant emission source can be determined by evaluating the ratio between different elements, which are called technogenic tracers (Calvo et al. 2013; Titos et al. 2014). The proportion between trace-metals such as Cu, Zn, Pb, and Cr, for instance, is a strong chemical marker for the origin of emissions, enabling segregation either between natural and anthropogenic sources (Weiping et al. 2014) or among different types of

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✉ Luzimar Campos da Silva
luzimar@ufv.br

[1] Department of Plant Biology, Universidade Federal de Viçosa, Av. PH Rolfs S/N, Viçosa, MG 36570-900, Brazil

[2] Research Center for Ecology, Instituto de Pesquisas Ambientais, Av. Miguel Stéfano 3687, Água Funda, Caixa Postal 68041, São Paulo, SP 04045-972, Brazil

[3] Department of Physics, Universidade Federal de Viçosa. Av. PH Rolfs S/N, Viçosa, MG 36570-900, Brazil

anthropogenic sources (Migliavacca et al. 2012; Khillare et al. 2014; Mohiuddin et al. 2014).

Fine particle deposition may cause contamination of soil and plant species (Schreck et al. 2011; Liu et al. 2019). Due to the large contact area of tree canopies and to foliar traits such as leaf morphology and surface roughness (Beckett et al. 2000; Sharma et al. 2020; Andrade et al. 2022), leaves can capture PM from the local atmosphere and thus be efficiently used to track air pollution. Several studies have suggested that PTE accumulation in tree leaves can be used as a reliable biomonitor to assess air quality. For instance, Monaci et al. (2022) determined the pattern of airborne PTE contamination across five different towns from central Italy using leaves of *Quercus ilex*. The contamination profile was mainly characterized based on the accumulation of Al, Ba, Hg, and Sb, which on the one hand reflected the geolithological settings of the area and on the other revealed the local anthropogenic disturbances (Monaci et al. 2022). In urban areas of Bojnourd, Iran, *Fraxinus excelsior* and *Pinus eldarica* were described as suitable tools for biomonitoring Pb and Zn, respectively (Solgi et al. 2020). Analogously, the high concentration of Ni, Pb, V, and Co in leaves of *Nerium oleander* and *Conocarpus erectus* reflected the influence of industrial activity on the metal content of plants from areas surrounding the industrial zone of Asaloyeh, Iran (Safari et al. 2018).

Located in southeastern Brazil, in a region called Steel Valley, Ipatinga city grew around its steel-industry park (Neves and Camisasca 2013). The city hosts within its urban area the largest industrial complex of flat-steel production in Latin America (Neves and Camisasca 2013), while also having one of the largest vehicle fleets in the country, with just over 160 thousand vehicles (Denatran 2021). Both these urban activities, i.e., industrial and vehicular, are known to emit large amounts of PM, which itself characteristically has particular metals adsorbed to its surface. Monitoring atmospheric conditions in the city is important to evaluate its air quality and to ultimately promote preventive actions concerning public health and environmental impacts. Plants of *Joannesia princeps* exposed in the city, especially on sites downwind to the steel mill, showed severe morphological and anatomical alterations in leaves and extrafloral nectaries, which has been speculated to have ecological consequences to plant–insect interactions (Silva et al. 2023). Daily annual mean of inhalable particles (PM_{10}) in Ipatinga city surpasses the limit established by the World Health Organization, of 20 $\mu g.m^{-3}$ (Programa Cidades Sustentáveis 2017). Thus, developing new strategies and approaches to constantly and more broadly assess air quality in the city should be encouraged.

Biomonitoring methods using plant material have some remarkable advantages, like the technological simplicity with which they enable the determination of sites with higher levels of PTE pollution (Arndt and Schweizer 1991). Seeking to determine the predominant PTE emission sources at Ipatinga, in the Brazilian Steel Valley, based on an evaluation of PM-associated PTEs, we aimed to perform a biomonitoring assay using two tropical legumes, *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya*, in order to assess chemical markers for the origin of emissions in the region, distinguishing between different anthropogenic sources.

Material and methods

Plant species and cultivation conditions

The studied species were *Paubrasilia echinata* (Lam.) Gagnon, H.C.Lima & G.P.Lewis and *Libidibia ferrea* (Mart. ex Tul.) L.P.Queiroz var. *leiostachya* (Benth.) L.P.Queiroz (Leguminosae–Caesalpinioideae). We selected these two species due to their both being native to the Atlantic forest and, most importantly, having distinct leaf morphologies and different epicuticular wax micromorphologies (Andrade and Silva 2017), both of which are leaf attributes that determine differential degrees of particle adsorption (Räsänen et al. 2013; Huang et al. 2015; Wang et al. 2015). Saplings were obtained from a nursery, transplanted, and acclimatized in a greenhouse, the procedure for which is described in detail in Andrade et al. (2022).

Study site

After acclimation, plants proceeded to the biomonitoring assay in four urban sites subjected to PM deposition in Ipatinga city and in one reference site at a neighboring park. One plant lot of each species remained in the greenhouse for analysis of leaf surface traits by cryoprocessing (see subsection "Cryoprocessing" below). In Ipatinga, plants were exposed at Bom Retiro (19° 30′ 42.2″ S, 42° 33′ 25.5″ W), Cariru (19° 29′ 28.8″ S, 42° 31′ 43.5″ W), Veneza (19° 28′ 20.1″ S, 42° 31′ 35.9″ W), and Cidade Nobre (19° 27′ 41.0″ S, 42° 33′ 37.2″ W) neighborhoods (Fig. 1). These four sites were selected for being those whose atmosphere is constantly monitored through analytical methods by automatic stations.

As reference station, an exposure rack was installed at the Rio Doce State Park plant nursery (19° 45′ 45.5″ S 42° 37′ 50.6″ W). The park was chosen as reference for being a well-preserved fragment of Atlantic rainforest that was near Ipatinga and presumably free, in its interior, of urban and industrial emissions from the Steel Valley Metropolitan Region (Andrade et al. 2022).

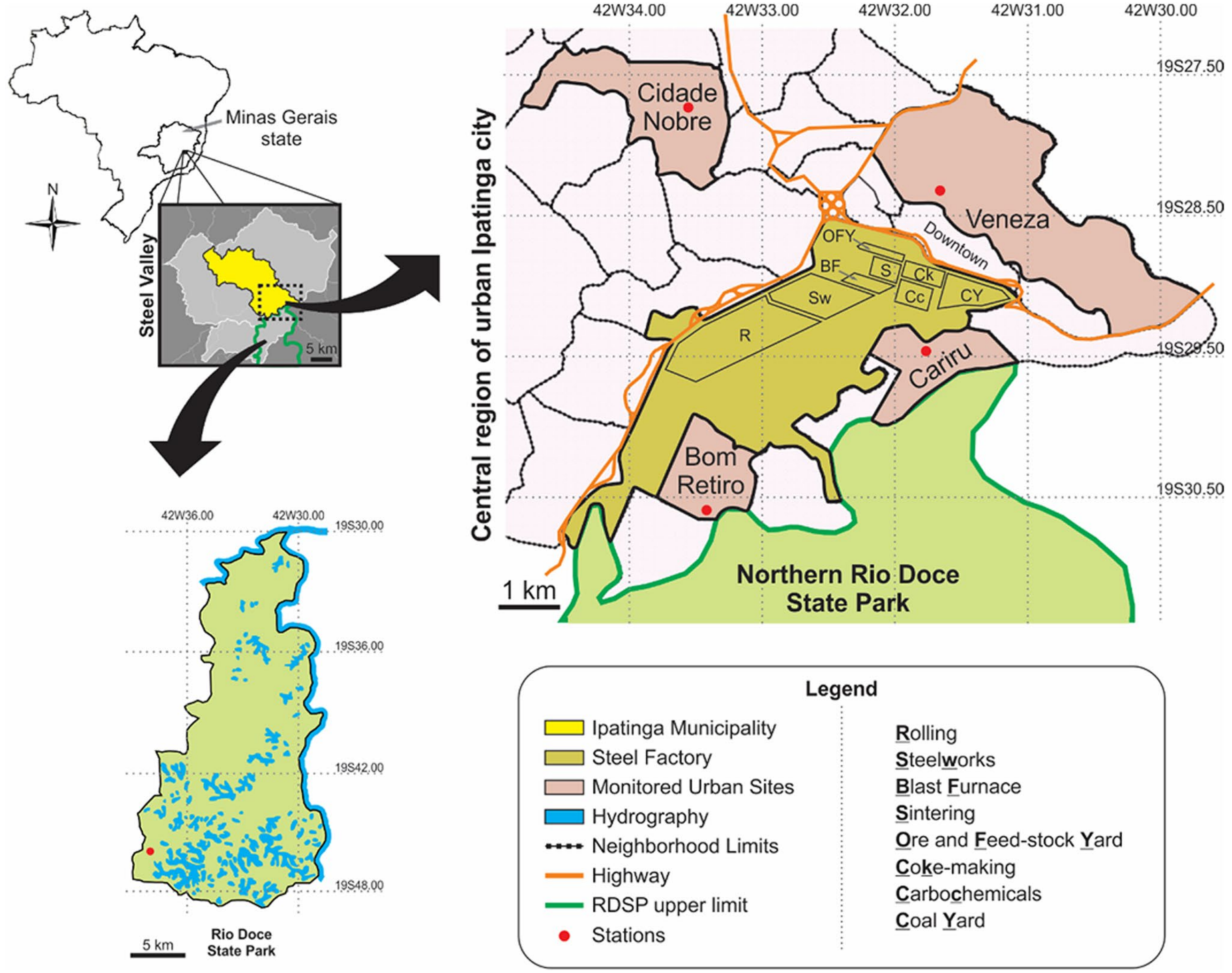


Fig. 1 Study area and sampled sites in Ipatinga city, southeastern Brazil, located in a metropolitan region called Steel Valley. The sampling sites were four neighborhoods around a steel factory: Bom Retiro, Cariru, and Veneza and a more distant one, Cidade Nobre. A site in the Rio Doce State Park, adjacent to the municipality, served as reference (adapted with permission from Andrade et al. 2022)

Experimental design and field exposure

Five potted individuals ($n = 5$ plants) of each species ($n = 2$ species) were exposed at each site ($n = 5$ sites) after random selection. The exposure procedures summarized here are detailed in Andrade et al. (2022). In the experimental period, plants were irrigated with deionized water to avoid interference by trace metals. Field exposure took place in the wet season, from October 2014 through January 2015, totalizing 90 days of exposure, the minimum period considered by some authors for carrying out an analytical monitoring in the context of environmental impact studies (Frondizi 2008).

Meteorological data and airborne particle concentration

Particle concentration (TSP: total suspended particles; PM_{10}: inhalable particles with aerodynamic diameter < 10 µm; $PM_{2.5}$: inhalable particles with aerodynamic diameter < 2.5 µm) in the Ipatinga atmosphere during the exposure period was obtained by beta particle attenuation, from the analytical monitoring performed by the city Air Quality Continuous Monitoring Automatic Network. The meteorological parameters wind direction, wind speed, air temperature, rainfall, atmospheric pressure, relative air humidity, and global solar radiation are also evaluated by the stations. All the above-mentioned variables (particle concentration and meteorological parameters) are assessed on an hourly basis by the stations. The biomonitoring racks were placed 2 to 3 m away from each of the four monitoring stations.

Metal contents in plant dry matter

Inorganic chemical elements that are usually associated with several emission sources were evaluated in plants from the four urban sites and from the reference site. The amounts of Cu, Fe, Zn, Mn, Ni, Pb, Cr, Ba, Al, Co, and V were assessed since they are technogenic tracers of steel industry and several types of vehicular emission, among others (Calvo et al. 2013), and the amounts of Ca and Mg were assessed since they are important components of slag (Shen et al. 2009) and could therefore be also used as tracers. Three out of the five replicates (plant individuals) were sampled for metal quantification, being chosen by simple random sampling.

In order to determine the concentrations of Cu, Fe, Zn, Mn, Ni, Pb, Cr, Ca, and Mg in the plant biomass, at the end of the exposure period, all leaves except those from the youngest and from the two oldest nodes (in the latter case, aiming to prevent soil contamination) were collected, oven-dried at 75 °C until constant weight and ground in a stainless steel willye-type knife mill (model Star FT 50, American Lab, Charqueada, Brazil). Then, 0.5 g of each sample was digested in 10 mL nitric-perchloric solution (nitric acid + perchloric acid, 4:1) (Sarruge and Haag 1974), heated until 200 °C, and the volume of the solution was then adjusted to 25 mL with deionized water. The elements were quantified by atomic absorption spectrometry (spectrometer model 240FS, Agilent Technologies, Santa Clara, USA).

For Ba, Al, Co, and V quantification, samples were also subjected to nitric-perchloric digestion, but analyzed by inductively coupled plasma optical emission spectrometry (spectrometer model Optima 8300, PerkinElmer, Waltham, USA). The blank sample used was the nitric-perchloric solution itself, with no addition of plant dry matter.

Enrichment factors

The enrichment factor (EF) represents the relative abundance of a given element in leaves of plants exposed at a certain presumably contaminated site compared against leaves of plants from a reference site. In order to determine to what degree were plant leaves enriched with metals, we calculated the EF of plants exposed at the urban sites by comparing metal concentration in plants from these sites against that of plants from RDSP, the reference site. The degree of enrichment of plant samples with each of the quantified metals was assessed using Eq. 1:

$$\mathrm{EF} = \frac{C\mathrm{site}}{C\mathrm{background}} \tag{1}$$

where EF = enrichment factor; C_{site} = metal concentration in plants from the urban sites; and $C_{\mathrm{background}}$ = metal concentration in plants from the reference site.

The threshold for considering plant samples enriched was $\mathrm{EF} > 2$, following Mingorance et al. (2007).

Cryoprocessing

Samples from unexposed plants (i.e., individuals that remained in the greenhouse after cultivation and were not exposed to any environmental conditions outside it; see subsection "Plant species and cultivation conditions") were analyzed through fracturing in liquid nitrogen for observation under scanning electron microscopy (SEM), aiming to characterize the leaf epidermal surface in cross-section at the high resolution provided by SEM. Fully expanded third-node leaf samples were collected from three randomly chosen individuals of the two species at sapling stage. Leaf fragments ca. 16 mm^2 were cut with a razor blade from the mid portion of pinnulae, which were taken from the mid-portion of pinnae, which in turn were obtained from the mid portion of the leaf. Samples were fixed in a solution of glutaraldehyde (2.5%) and paraformaldehyde (10%) (Karnovsky 1965) in a 0.1 M sodium cacodylate buffer (pH 7.2) for a minimum period of 24 h, washed in that same buffer, and immersed for 1 h in 30% glycerol, which acts a cryoprotector. Then, samples were washed in deionized water, enveloped in silk paper, immersed in liquid nitrogen, placed onto a metal support, and cut into smaller fragments (ca. 8 mm^2) with a scalpel (F.A.O. Tanaka, personal communication, after F.C. Miguens; modified). Leaf fragments were once again washed in deionized water, dehydrated in acetone, critical-point dried with liquid CO_2 (critical point drier model CPD 030, Bal-Tec, Balzers, Liechtenstein), sputter-coated with gold (sputter coater model FDU 010, Bal-Tec), and then, visualized and photographically documented in a scanning electron microscope (model LEO 1430 VP, Carl Zeiss, Jena, Germany) through detection of secondary electron signals at 15 kV.

Statistical analysis

Data on metal accumulation were subjected to two-way analysis of variance (ANOVA) followed by Tukey's test at 5% probability, multivariate analysis of variance (MANOVA), and principal component analysis (PCA). The analyses were performed using R-software version. 4.0.4 (R Development Core Team 2021).

Results

Meteorological data and airborne particle concentration

During the experimental period, the heaviest weekly rainfall was registered at Bom Retiro (96.0 mm). High temperatures characterized the entire period, with maximum temperature ranging between 30 and 40 °C. Average values of mean temperature were 24.74 °C at Bom Retiro, 27.11 °C at Cariru, 26.67 °C at Veneza, and 26.44 °C at Cidade Nobre (Fig. 2).

Considering all monitoring stations in Ipatinga city, the predominant wind was the northeast one. Much occasionally were there discordant patterns. For example, there were weak, infrequent southeast winds at Bom Retiro and

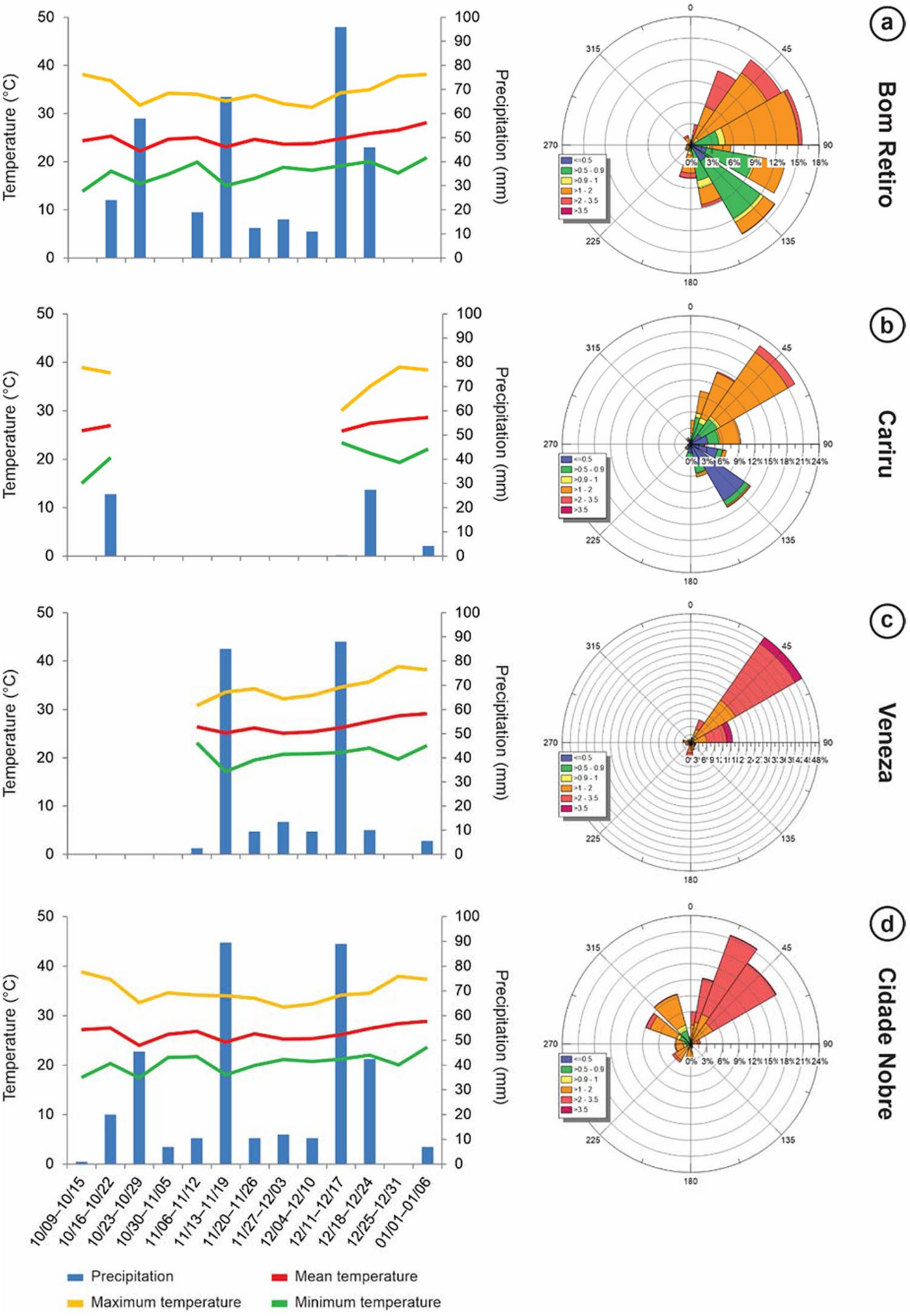


Fig. 2 Meteorological characterization of the studied sites. Climatograms (left column) showing weekly accumulated precipitation (mm) and mean temperature (°C), and windrose charts (right column) showing predominant wind speed and direction in the central region of Ipatinga city along the 13-week exposure period. Values not shown correspond to readings not registered by the automatic stations. **a** Bom Retiro. **b** Cariru. **c** Veneza. **d** Cidade Nobre

analogously low, infrequent northwest ones at Cidade Nobre (Fig. 2).

Airborne PM, to which the evaluated metals mostly were adsorbed, had their emission dynamics in the region evaluated by Andrade et al. (2022). According to the authors, the pattern of emissions for total suspended particles (TSP) was Cariru (40.98 μm m^{-3}) > Veneza (37.83 μm m^{-3}) > Bom Retiro (35.36 μm m^{-3}) > Cidade Nobre (32.17 μm m^{-3}), whereas for inhalable particles (PM_{10}), the pattern was Cariru (25.52 μm m^{-3}) > Cidade Nobre (22.74 μm m^{-3}) > Bom Retiro (22.21 μm m^{-3}) > Veneza (17.46 μm m^{-3}).

Metal contents in plant dry matter

Among the investigated tracers, vanadium was not detected in any samples and, therefore, is not shown in the data summarized in Table 1.

There were significant interactions in the accumulation of Ca, Cu, Fe, Mn, Ni, Pb, and Co between the factors species and exposure site. *Libidibia ferrea* var. *leiostachya* showed higher Ca contents in plants at the reference station in comparison with plants exposed at Cariru. This species accumulated higher Cu contents than *P. echinata*. Increased Cu amounts were detected at Bom Retiro, Cariru, and Cidade Nobre. The opposite was observed with Fe, Ni, and Pb accumulation. *P. echinata* accumulated higher contents of these three metals than *L. ferrea* var. *leiostachya* at Bom Retiro and higher contents at this neighborhood than at the other sites (with the exception of Pb at Veneza). *L. ferrea* var. *leiostachya* showed higher Mn contents in plants at RDSP than in plants exposed at Cariru and higher contents than *P. echinata* at the former site. Ni contents followed the inverse pattern, being higher in plants at Cariru than in plants at RDSP. As for Co, *L. ferrea* var. *leiostachya* accumulated higher amounts than *P. echinata* at Cariru and Cidade Nobre. The sites Cariru, Veneza and Cidade Nobre provided higher Co accumulation to plants of *L. ferrea* var. *leiostachya* than did Bom Retiro and RDSP (Table 1).

Overall, besides Fe, increasing concentrations along the wind direction were found with elements Zn, Mn, Ni, Pb, and Cr, as well as particularly higher amounts of Al. Plants of both species exposed at Bom Retiro showed the highest contents of these elements (Table 1).

Aiming to explore the variation between species and among exposure sites, principal component analysis was performed with data on elemental quantification in order to obtain a small number of linear combinations of the 12 metals which accounted for most of the variability in the data. Three components were extracted, with eigenvalues higher than one. Together, they accounted for 80.24% of the variability in the data.

Considering the two principal components plotted, component 1 was composed by Cr, Al, Ni, Pb, Fe, and Ba, and component 2 by Zn, Mg, Mn, Cu, Co, and Ca (Fig. 3).

The two species were clearly separated by PCA, forming two well-defined clusters. Associations between the variation of certain metals could also be noticed. Fe and Pb varied together, and so did Al and Ni; Co, Cu, and Mg; and Ca, Mn, and Zn. Barium varied singly toward *P. echinata* data, and Cr varied singly in the verge between species (Fig. 3).

Exposure sites were also clearly separated. The reference station, for instance, formed a well-defined cluster that was well segregated by component 1, showing the lowest component weights (Fig. 3).

Most elements varied toward the urban exposure stations. The element that varied more closely toward the reference station was Mg (Fig. 3).

Both components influenced the result. Component 1, however, separated species among sites, according to pollution level, while component 2 did such separation to a lesser degree. Component 1 seems, therefore, to be a revealer of pollution, whereas component 2 seems to discriminate between species (Fig. 3).

Enrichment factors

Samples were considered enriched when having EF > 2, following Mingorance et al. (2007). In that sense, of all elements quantified, Fig. 4 shows only those that were enriched in plants from at least one studied site. Of the analyzed elements, EF was > 2 for Fe, Al, Ni, Cr, and Ba in *P. echinata* and Fe, Cu and Co in *L. ferrea* var. *leiostachya* (Fig. 4; Table 2).

All elements showed highest enrichment at Bom Retiro (with the exceptions of Ba in *P. echinata* and Co in *L. ferrea* var. *leiostachya*). In both species, Fe was the element with which plants were enriched the most. Fe was also the only enriched element that the two species had in common. In *P. echinata*, the Fe enrichment factor was more than double the one in *L. ferrea* var. *leiostachya*. In both species, plants were least enriched with Fe at Cidade Nobre. Fe, the main technogenic tracer for the industrial activity taking place in the region (steelmaking), showed the following patterns of enrichment: Bom Retiro > Veneza > Cariru > Cidade Nobre, in *P. echinata*, and Bom Retiro > Cariru > Veneza > Cidade Nobre, in *L. ferrea* var. *leiostachya* (Fig. 4).

Foliar structure (cryoprocessing)

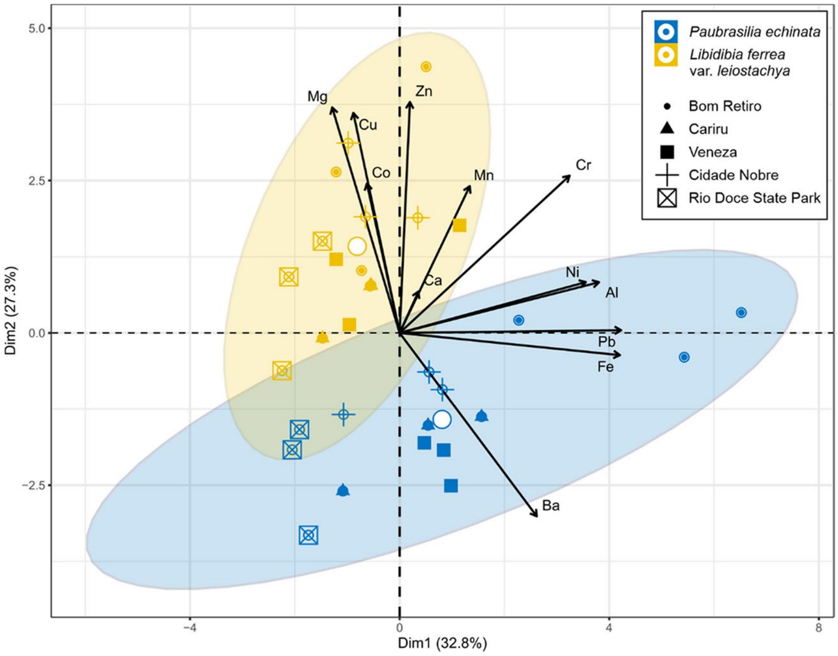
Foliar epidermal relief has highly contrasting traits between the two species, which enables a differential rate of particle retention by each of them. While *P. echinata* has a smooth layer of waxes covering the leaf surface and no conspicuous grooves, *L. ferrea* var. *leiostachya* has a rough surface

Table 1 Analysis of variance of trace-metal accumulation in *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya* leaves after 3 months of exposure to urban pollution from a steel-mill pole in southeastern Brazil

Species (Sp)	Site	Metal▲											
		Ca	Mg	Cu	Fe	Zn	Mn	Ni	Pb	Cr	Ba	Al	Co
Paubrasilia echinata	Bom Retiro	1.820 ± 0.175^{Aa}	0.178 ± 0.024^{A}	5.767 ± 0.153^{Ba}	$1474.500 \pm 597.995^{Aa}$	47.133 ± 4.801	545.800 ± 25.982^{Aa}	3.667 ± 0.737^{Aa}	13.000 ± 0.721^{Aa}	3.033 ± 0.289^{a}	3.816 ± 1.233	109.834 ± 51.816^{a}	0.092 ± 0.026^{Aa}
	Cariru	1.640 ± 0.431^{Aa}	0.156 ± 0.009^{A}	5.367 ± 1.365^{Ba}	703.900 ± 297.616^{Ab}	34.067 ± 8.264	458.433 ± 148.647^{Aa}	1.990 ± 0.264^{Ab}	11.100 ± 0.458^{Ab}	1.967 ± 0.305^{ab}	4.601 ± 2.116	57.245 ± 16.741^{ab}	0.116 ± 0.037^{Ba}
	Veneza	1.477 ± 0.216^{Aa}	0.159 ± 0.024^{A}	5.600 ± 0.529^{Aa}	721.267 ± 21.371^{Ab}	28.800 ± 1.819	422.533 ± 17.264^{Aa}	2.000 ± 0.1^{Ab}	11.300 ± 0.360^{Aab}	1.833 ± 0.252^{ab}	4.765 ± 1.343	79.514 ± 13.392^{ab}	0.116 ± 0.014^{Aa}
	Cidade Nobre	1.988 ± 0.213^{Aa}	0.179 ± 0.003^{B}	6.400 ± 1.082^{Ba}	465.900 ± 62.085^{Ab}	38.200 ± 1.778	413.033 ± 25.022^{Aa}	1.800 ± 0.436^{Ab}	10.900 ± 1.212^{Ab}	2.367 ± 0.586^{ab}	2.904 ± 0.028	71.437 ± 3.681^{ab}	0.086 ± 0.028^{Ba}
	Rio Doce State Park	1.647 ± 0.233^{Aa}	0.142 ± 0.027^{B}	4.833 ± 0.586^{Aa}	255.767 ± 20.476^{Ab}	32.517 ± 5.858	328.800 ± 39.392^{Ba}	1.400 ± 0.1^{Ab}	10.267 ± 0.153^{Ab}	1.467 ± 0.416^{b}	2.306 ± 1.418	39.044 ± 8.887^{b}	0.096 ± 0.020^{Aa}
Libidibia ferrea var. *leiostachya*	Bom Retiro	1.623 ± 0.978^{Aab}	0.303 ± 0.067^{A}	15.167 ± 1.101^{Aa}	633.533 ± 73.559^{Ba}	60.150 ± 15.368	$596.600 \pm 310.418^{Aab}$	1.933 ± 0.115^{Bab}	10.733 ± 0.472^{Ba}	2.433 ± 0.513^{a}	0.398 ± 0.382	67.648 ± 3.148^{a}	0.133 ± 0.027^{Ab}
	Cariru	0.640 ± 0.076^{Ab}	0.186 ± 0.019^{A}	12.033 ± 0.153^{Aa}	364.833 ± 38.317^{Aa}	40.700 ± 3.315	298.833 ± 79.442^{Ab}	2.667 ± 0.252^{Aa}	10.833 ± 0.378^{Aa}	1.933 ± 0.115^{a}	0.389 ± 0.099	60.933 ± 4.902^{a}	0.201 ± 0.027^{Aa}
	Veneza	1.780 ± 0.809^{Aab}	0.232 ± 0.084^{A}	8.167 ± 1.686^{Ab}	335.867 ± 74.423^{Aa}	44.433 ± 5.529	$601.367 \pm 187.273^{Aab}$	2.067 ± 0.153^{Aab}	10.833 ± 0.503^{Aa}	2.500 ± 0.693^{a}	0.793 ± 0.454	67.490 ± 17.660^{a}	0.172 ± 0.0388^{Aa}
	Cidade Nobre	1.463 ± 0.353^{Aab}	0.329 ± 0.081^{A}	11.233 ± 2.892^{Aa}	298.900 ± 9.853^{Aa}	44.117 ± 7.447	$657.167 \pm 148.806^{Aab}$	2.133 ± 0.321^{Aab}	11.033 ± 0.814^{Aa}	2.433 ± 0.115^{a}	0.987 ± 0.622	76.332 ± 19.414^{a}	0.233 ± 0.014^{Aa}
	Rio Doce State Park	2.642 ± 0.824^{Aa}	0.302 ± 0.069^{A}	6.700 ± 2.107^{Ab}	239.267 ± 40.457^{Aa}	52.667 ± 11.481	805.300 ± 274.614^{Aa}	1.533 ± 0.153^{Ab}	10.167 ± 0.305^{Aa}	1.967 ± 0.451^{a}	0.702 ± 0.287	39.145 ± 5.820^{a}	0.094 ± 0.016^{Ab}
Analysis of variance													
SV	DF	Mean square											
Sp	1	$0.05393^{n.s}$	0.086941***	192.533***	917,630***	1129.15***	187,546*	$0.05633^{n.s}$	2.6403*	$0.10800^{n.s}$	$287,463^{n.s}$	$621.77^{n.s}$	0.031948***
Site	4	$0.77018^{n.s}$	$0.006838^{n.s}$	20.094***	561,481***	279.74**	$37,278^{n.s}$	1.41967***	2.0550**	0.93217**	$113,996^{n.s}$	2105.30**	0.004966***
Sp×Site	4	0.88444*	$0.004505^{n.s}$	14.206**	145,174*	$54.79^{n.s}$	83,121*	1.38300***	1.3853*	$0.37050^{n.s}$	$113,928^{n.s}$	$580.24^{n.s}$	0.004688**

Means ± standard deviation. ▲Ca and Mg in dag kg^{-1}; other elements in mg kg^{-1}. ***Significant by *F* test ($P < 0.001$). **Significant by *F* test ($P < 0.01$). *Significant by *F* test ($P < 0.05$). *n.s.*, non-significant by *F* test ($P > 0.05$). Means followed by different letters differ by Tukey's test at 5% probability. Upper case letters compare species in a same site. Lower case letters compare sites in a same species

Fig. 3 Principal component analysis biplot of trace-metal accumulation in *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya* leaves after 3 months of exposure to urban pollution from a steel pole in southeastern Brazil



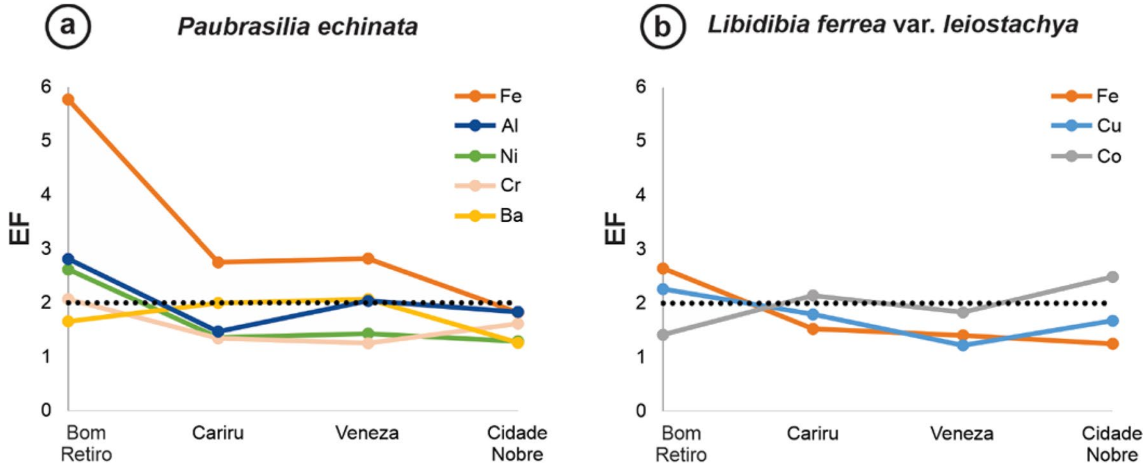


Fig. 4 Mean enrichment factor (EF) of plant leaves of *Paubrasilia echinata* (**a**) and *Libidibia ferrea* var. *leiostachya* (**b**) after 3 months of exposure to urban pollution from a steel pole in southeastern Brazil, for elements having EF > 2 in relation to plants from the reference site. The horizontal line represents the threshold of EF = 2, above which plants were considered enriched (following Mingorance et al., 2007)

with waxes deposited in the form of vertical platelets, and pronounced grooves at the anticlinal-wall region (Fig. 5). Roughness values for the leaf surface of these two species can be found in the study performed by Andrade et al. (2022).

Discussion

The predominance of northeast winds in the region initially suggested that Cidade Nobre would be the least impacted urban site and that Bom Retiro would be the most impacted one, particularly by gaseous pollutants and, among particulate matter, by the ones with lowest aerodynamic diameter (Marris et al. 2012; Almeida et al. 2015). As PM_{10} and $PM_{2.5}$ are particles with lower deposition rates, they tend to remain in the atmosphere for a longer period of time and be transported to further distant regions (Riffault et al. 2015; Garg and Sinha 2017; Jain et al. 2021). These predictions, however, were only partially met.

As shown by the atmospheric characterization through analytical monitoring and confirmed through the biomonitoring (Andrade et al. 2022), the site with highest amounts

Table 2 Enriched elements and probable sources of enrichment in the leaf dry matter of *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya* plants, after 3 months of exposure to urban pollution from a steel pole in southeastern Brazil, for elements having enrichment factor (EF) > 2 in relation to plants from the reference site. Enriched elements are listed in inverse order of magnitude, at each site, from highest to lowest EF

	Paubrasilia echinata		*Libidibia ferrea* var. *leiostachya*	
Site	EF > 2	Probable sources	EF > 2	Probable sources
Bom Retiro	Fe > Al > Ni > Cr	Vehicular and industrial	Fe > Cu	Vehicular and industrial
Cariru	Fe	Vehicular and industrial	Co	Industrial
Veneza	Fe > Ba > Al	Vehicular and industrial	-	-
Cidade Nobre	-	-	Co	Industrial

References: Lim et al. 2007; Almeida et al. 2009; Canha et al. 2012; Malizia et al. 2012; Calvo et al. 2013; Norouzi et al. 2015; Wang et al. 2017; Jia et al. 2018; Ali et al. 2019; Karmakar and Padhy 2019; Liang et al. 2023

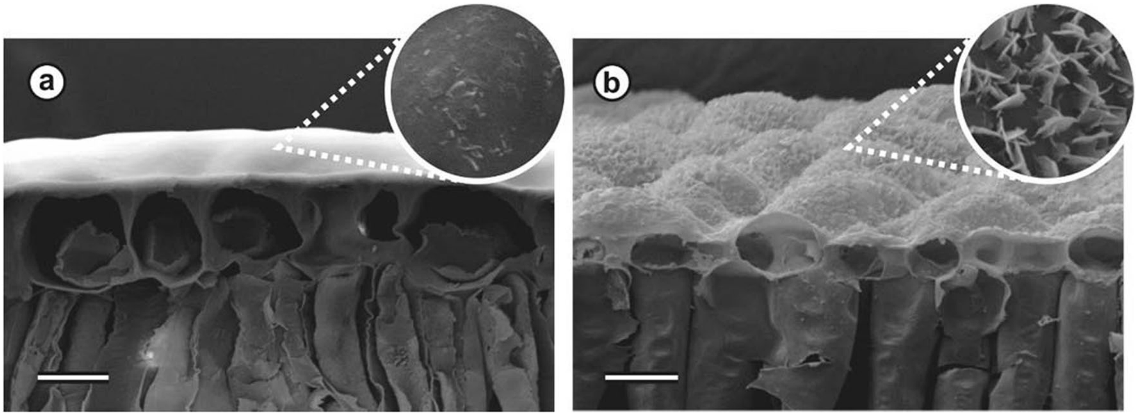


Fig. 5 Surface and cross-section of leaves of *Paubrasilia echinata* (**a**) and *Libidia ferrea* var. *leiostachya* (**b**), the species used in the biomonitoring of airborne particulate matter pollution in Ipatinga city, southeastern Brazil. Note the epicuticular waxes deposited in the form of a smooth layer in *P. echinata* and in the form of densely aggregated rosettes of vertical platelets in *L. ferrea* var. *leiostachya*. Bars = 10 µm

of PM_{10} was Cariru. As for large particles (PM_{100}), their accumulation by plants in the biomonitoring assay was quite homogenous among the evaluated sites ($F_{4,167} = 6.98$; $P > 0.001$), with the exception of Cariru, where more particles were adsorbed by the plants (Andrade et al. 2022). Cariru is the closest site to the coke-making, carbochemical, and coke-yard plants, being located south of them, along the northeast windstream. Yet, the second highest amounts were detected at Veneza, only then followed by Bom Retiro and Cidade Nobre. Veneza is also close to the coke-making, carbochemical, and coke-yard plants, as well as to the sintering plant and to the ore and feed-stock yard, but unlike Cariru, it is situated against the windstream. Thus, other possible sources of contamination at Veneza, including from the neighboring municipality, where there is a dumping ground nearby, should be investigated. In addition, Cidade Nobre was the most affected site by inhalable particles, after Cariru, followed by Bom Retiro and Veneza. Wind direction renders it little probable that Cidade Nobre is affected by steel industry activity. Instead, vehicular emissions seem to be the predominant factor in this neighborhood. The source of $PM_{2.5}$ emissions is known to be mostly anthropogenic (Ottelé et al. 2010; Liu et al. 2020).

P. echinata showed higher accumulation of Al, Ni, Pb, Fe, and Ba, while *L. ferrea* var. *leiostachya* accumulated higher amounts of Mg, Cu, Co, Zn, Mn, and Ca. Only Cr could not be segregated by neither principal component. It would seem like *L. ferrea* var. *leiostachya* leaves tend to retain Cu and Co particles, whereas *P. echinata* leaves tend to retain Fe, Ni, Pb, Al, and Ba particles. Elemental enrichment results also support this hypothesis, as the two species were enriched with different elements. These differences between species could have presumably arisen either from soil or from the leaf surface structure through some mechanism of adhesion of specific types of particles.

Andrade et al. (2022) reported that *L. ferrea* var. *leiostachya* retained lower amounts of particles due to surface traits of its leaves that confer them higher self-cleaning effect, like the higher roughness given both by the convex outer periclinal wall of epidermal cells and by its wax-layer microsculpturing. It is intriguing that only essential micronutrients (Fe, Cu, and Co) were enriched in *L. ferrea* var. *leiostachya*. Liu et al. (2019) reported that newly deposited metals from the atmosphere may be preferentially retained in topsoil and be highly bioavailable for plant absorption (Liu et al. 2019). This would suggest that the Fe, Cu, and Co enrichment in *L. ferrea* var. *leiostachya* took place from the atmosphere-soil-root transfer pathway, thus leading us to discard direct foliar deposition of PM and associated PTEs as the main route. On the other hand, the opposite was found in *P. echinata*, and, for that reason, along with its higher potential for accumulating PM on its leaves (Andrade et al. 2022), this species is better recommended for biomonitoring air pollution as a bioaccumulator.

Despite the fact that Cariru was the site with highest amount of PM, as shown by both analytical monitoring and biomonitoring (Andrade et al. 2022), Bom Retiro was the most impacted neighborhood by iron-containing particles, as plants from this site were the ones that were most enriched with Fe. Plants exposed at Bom Retiro showed mean iron contents in the plant dry matter of *P. echinata* (1475 ± 598 mg kg^{-1}) and *L. ferrea* var. *leiostachya* (634 ± 74 mg kg^{-1}) higher than the maximum iron contents found in plants of *Eugenia uniflora* (895 mg kg^{-1}) and *Clusia hilariana* (596 mg kg^{-1}) exposed in a seven-month active biomonitoring assay at the vicinities of an iron ore pelletizing factory from the Brazilian southeastern coast (Silva et al. 2015). These values, especially of *P. echinata*, were also higher than the maximum iron contents found in the bromeliad *Tillandsia usneoides* after 45 days of exposure in an active biomonitoring in the surroundings of the collapsed tailings dam in Brumadinho, southeastern Brazil (758 mg kg^{-1} at Córrego do Feijão and 641 mg kg^{-1} at Parque da Cachoeira) (Parente et al. 2023). The analysis of PTEs in PM is a safe method for assessing source-related emissions (Almeida et al. 2015; Riffault et al. 2015; Liu et al. 2018b). Fe is a technogenic tracer of emissions from iron and steel industries (Calvo et al. 2013), as iron particles are usually a product of basic oxygen steel-making and sintering (Almeida et al. 2009; Canha et al. 2012; Jia et al. 2018). Although the steelworks and sintering plants are nearer Cariru, the highest iron accumulation in plants exposed at Bom Retiro was probably related to the predominant wind direction (northeast) in the region, which transports airborne particles to the southwest, where Bom Retiro is located. In a study on the effects of urban and industrial pollution form Ipatinga on leaves and extrafloral nectaries of *Joannesia princeps*, plants exposed at Cariru and Bom Retiro were the most affected (Silva et al. 2023). *Joannesia princeps* has also been suggested to be a biosensor of the impact by acid precipitation, a phenomenon which has been detected in some urban Brazilian areas within the Atlantic forest domain (Andrade et al. 2020).

The degree of enrichment by elements into the magnetic particles that are deposited and accumulated on plant leaves and other biological receptors can indicate the main emission sources of air pollutants, such as vehicular and industrial activities (Winkler et al. 2019; Rohra et al. 2023). The high EFs of Fe and the other elements (Ni, Al, Cr, Ba, Co, and Cu) suggest a dominance not only of industrial activity but also of vehicular traffic as the sources of air pollution in the studied region. The major contributors to vehicular emissions of these elements are road surface abrasion, resuspension of road dust, wear of vehicular components, tire clutch, and brake wear, while industrial emissions contribute mainly through fuel combustion (coal, oil, and coke), furnace, and gas turbines (Ali et al. 2019). Some PTEs, like Ba, Cr, Fe, Ni, and Cu, are present in fuels and lubricating oils as additives and are also originated from industrial metallurgical processes (Lim et al. 2007; Malizia et al. 2012; Calvo et al. 2013; Norouzi et al. 2015; Wang et al. 2017; Karmakar and Padhy 2019; Liang et al. 2023).

An important following step to the present research would be to perform a passive biomonitoring using the adult individuals already planted throughout the city. Particularly *P. echinata* showed to be promising for this purpose and, thereby, evaluating the trees already in the city which are constantly exposed to local pollution would raise complementary information to the results herein. In addition, *P. echinata* is extensively used as ornamental not only in Ipatinga city but also in several other Brazilian cities (Rocha and Barbedo 2008; Moro and Castro 2015; Oliveira et al. 2019), due mainly to its historical significance, as it is the national symbol-tree of the country (von Muralt 2006). Standardization remains one of the main issues with comparability of passive biomonitoring studies (Tarricone et al. 2015; Corada et al. 2021). Thus, elaborating standardized approaches with this species, in alignment with international studies, and investigating its potential for passive biomonitoring might allow for future establishment of a monitoring network that could sample several cities across the country and give good, comparable results, following high international standards.

Conclusions

In Ipatinga, Cariru is the neighborhood subjected to highest loads of total suspended particles and inhalable particles, due mostly to its proximity to the coke-making, carbochemical, and coke-yard plants nearby. However, Bom Retiro is the neighborhood where the exposed individuals of *Paubrasilia echinata* and *Libidibia ferrea* var. *leiostachya* accumulated highest amounts of Fe. This may be due mainly to the predominant wind direction in the region, northeast, which places Bom Retiro downwind to the local steel industry. At Veneza, other possible sources of contamination, including a nearby dumping ground from the neighboring municipality, should be further investigated. Plants were enriched the least with Fe at Cidade Nobre, which however was the site most affected by inhalable particles, mainly due to vehicular emissions. While Fe was the main technogenic tracer for the industrial activity taking place locally (steelmaking), other elements like Ni, Al, Cr, Ba, and Cu indicate the dominance not only of industrial activity but also of vehicular traffic as the sources of air pollution in the region, which originated chiefly from road surface abrasion, resuspension of road dust, wear of vehicular components, tire clutch, and brake wear.

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Data availability The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

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