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Research Paper

Multivariate analysis of metals in particulate matter of atmospheric air from Minas Gerais, Brazil: high metal concentrations

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ABSTRACT

Considering the importance of the evaluation of air pollutants in areas with higher population flow and that the particulate material is usually an air pollutant associated with health problems, this work aimed to determine the concentrations of particulate matter in two distinct fractions: the Total Suspended Particles (TSP) and Particulate Matter with diameter less than 2.5 µm ($PM_{2.5}$). It also aimed to evaluate the concentrations of twenty-one (21) chemical elements present in particulate matter and conducted a study of multivariate analysis. Samplings were performed from October to December, 2013 in the urban area of Montes Claros in Minas Gerais in sites identified as central, industrial and control areas. The sampling method used for particulate matter collection was the Large Volume Sampler and the determination of chemical elements was performed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The results for $PM_{2.5}$ and TSP are below the limits recommended by major environmental legislations. However, the concentration of some chemical elements are above the recommended values, highlighting Barium (Ba), zinc (Zn) nickel (Ni) and vanadium (V) in central and industrial area samples. Some similarities were also observed between sampling sites and the concentrations of chemical elements showing regularities and common origin between samples in the studied areas.

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Key words: Metals, particulate matter, TSP, $PM_{2.5}$, air quality.

INTRODUCTION

Air pollution follows humanity since ancient times. However, it has intensified as a result of the Industrial and Revolution (Braga et al., 2005). Technological progress, industrial development and population growth resulted in large amounts of emissions of both gaseous pollutants and particulate matter (PM) considerably increased issues related to environmental degradation (Kelly and Fussell, 2012; Cassee et al., 2013; Munir et al., 2016). Such air pollutants are mostly mainly formed by gaseous substances and vapors and particulate matter has been the subject of numerous studies and regulations for emissions control due to the impacts they cause on the environment (Vahlsing and Smith, 2012; Cakmak et al., 2014).

Among the air pollutants, the PM is the most studied (Mariani and Mello, 2007; Arbex et al., 2012) being usually a complex system containing liquid and solid particles with aerodynamic diameters in the range 0.01 to 100 µm according to Environmental Protection Agency-United States (EPA-US). The inhalable particulate material with an aerodynamic diameter not exceeding 10 µm is divided into two categories: the fine fraction, with a size up to 2.5 µm, $PM_{2.5}$ and coarse fraction with particles having diameters between 2.5 and 10 µm, PM_{10} (Baird and Cann, 2011; Marques and Santos, 2012).

In recent years, there has been increasing interest in the study on concentrations of particulate matter at the fractions PM_{10} and $PM_{2.5}$. Recent researches showed that the exposure to these particles cause severe damage to human health in short and long term (Ebert et al., 2012; Griffin, 2013; Rodriguez-Cotto et al., 2014; Yang et al., 2014). The exposure to fine particles have been consistently associated with serious health problems (World Health Organization, 2005; Pascal et al., 2014) being responsible for the incidence of respiratory, cardiovascular diseases and cancer (Dockery and Pope, 1994; Schwartz et al., 1996; Chan et al., 2001; Cançado, 2006; Krewski Samet, 2007; Moura et al., 2008) and in a small scale with adverse outcomes in the pregnancy process (Gouveia et al., 2004; Saldiva et al., 2013).

In Brazil, the environmental law that regulates the emission of particulate matter in the air is the National Council for the Environment (CONAMA) 03/1990 resolution. It currently determines parameters of air quality to be monitored in place with quality standards for total suspended particles (TSP) and particles with a diameter between 2.5 and 10 µm (CONAMA, 1990; FEAM, 2011). However, the World Health Organization (WHO), the EPA-US, as well as the environmental laws of the European Union countries recommended a standard of air quality for $PM_{2.5}$. In Brazil, several studies focused on monitoring the air quality in different regions and adopt the international laws along with the CONAMA resolution as standards.

The significant increase in environmental pollution is due to the increasing use of land transport powered by fossil fuels since they contribute to emissions of several pollutants and trace metals (Cassee et al., 2013; Weinbruch et al., 2014). Vehicles are considered to be substantial sources of metal through the wear pads and brake discs, rubber tires, gasoline and lubricating oil spill in urban areas or in areas affected by traffic. Possibly, in vehicles, the zinc origin is the result of wear in components of the brakes, tires and lubricating oils. Cadmium and copper are mainly from the brake wear.

Reported concentrations vary widely between the manufacturer and the type of coating and in some cases, several orders of magnitude. However, iron (Fe), copper (Cu), lead (Pb) and zinc (Zn) appear constantly and reports exhibit high concentrations in brake linings (Thorpe and Harrison, 2008; Grass et al., 2010). The road surface particles re-suspension also seems significant, particularly in dry climate regions (Abu-Allaban et al., 2003; Charron et al., 2007).

Although the road powder is mainly composed of mineral particles, it is also enriched with toxic metals. At present, road traffic is a major source of metal and particulate matter emissions, being used as chemical markers to identify emission sources (Amato et al., 2009).

Based on the aforementioned information, the objectives of this study were to determine the particulate matter concentrations in the region of Montes Claros, Minas Gerais, Brazil in two fractions, TSP and $PM_{2.5}$. It also aimed to evaluate the concentration of chemical elements present in the particulate material and conduct a multivariate analysis of the metal values found.

MATERIALS AND METHODS

The study area

Montes Claros is located in the North of Minas Gerais, Brazil and it has an estimated population of 390.212 inhabitants. However, its coverage area in the northern region of Minas is 128 602 km^2, where 1.63 million inhabitants dwell (IBGE, 2014). Samplings of particulate matter were collected in the urban area of Montes Claros and the sampling sites strategically defined as: industrial, central and control areas (Figure 1 and Table 1). Sampling took place between the months from October to December, 2013.

Sampling and chemical characterization

The sampling method adopted was the one that uses Large Volumes Samplers of glass fiber filters for TSP and $PM_{2.5}$ measurements, according to the rules of the Brazilian Association of Technical Standards (ABNT) - Brazilian standard NBR 9547/1997 and NBR 13412. The samples were collected monthly for periods of 24 h each. To determine the concentration of particulate matter (TSP and $PM_{2.5}$), the filter was placed in the desiccator for 24 h before being weighed. This procedure was repeated before and after sample collection. All weights were made in analytical scale (Shimadzu Scale Model AUX220, accuracy of 10 mg). The volume of sampled air corrected to standard conditions (25°C and 760 mmHg) was determined from the work flow and sampling time. A corresponding fraction to 25% of its area of each filter was withdrawn for extraction of particulate matter and chemical characterization according to IO-3.1 EPA-1999 method (EPA, 1999). The extraction of particulate matter was held in the microwave oven (CEM Corporation brand, MARS System 6 model) according to 3051 EPA-US method already optimized and installed at the equipment.

For the decomposition step, 10 ml of an acidic solution was added consisting of HNO_3 and HCl in the ratio 1: 3; from the extraction, 1 ml of the residual solution was taken and diluted to 10 ml with ultrapure water, 18 MΩcm, Falcon tube type. This procedure was also adopted for blank filters (without exposure filter). Subsequently, samples were taken to determine the concentration of chemical elements. The solutions were prepared with high purity water (18 MΩcm). The nitric and hydrochloric acids were distilled at sub-boiling system from acids with

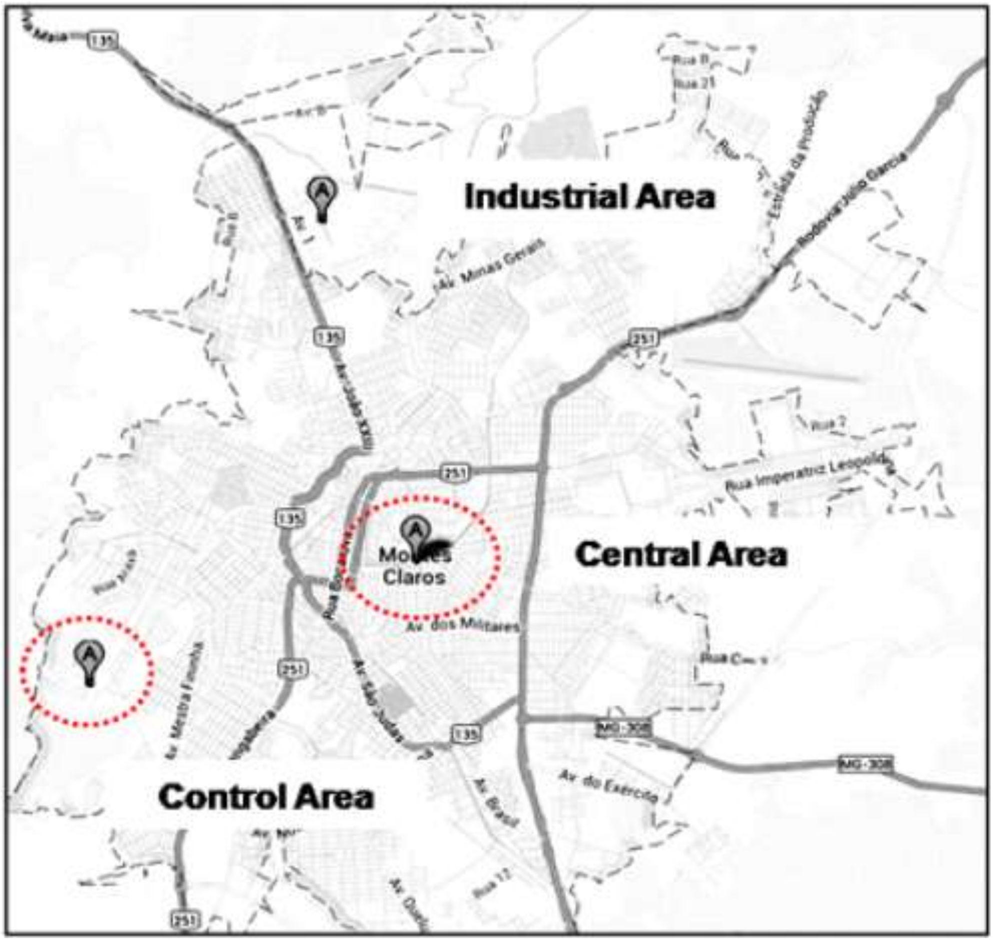

Figure 1: Sampling sites location in Montes Claros, Minas Gerais, Brazil.

Table 1: Sampling sites coordinates in Montes Claros, Minas Gerais, Brazil.

Sampling stations	Co-ordinates
Industrial area	16°68'949" south latitude and 43º87'138" west longitude
Central area	16°72'302" south latitude and 43º86'708" west longitude
Control area	16°44'575" south latitude and 43º54'138" west longitude

analytical grade.

The concentration of twenty-one (21) chemical elements (Al, Ba, Cd, Co, Cu, Mn, Rb, Mg, Tl, Pb, Ni, Se, Zn, V, U, Be, Bi, As, Cr, Ag and Fe) was conducted using an inductively coupled plasma mass spectrometer (ICP-MS), ELAN DRCII Perkin-Elmer model, with the operating conditions described (Table 2).

Statistical analysis

The results were presented using descriptive statistics for the concentrations of particulate material and chemical characterization. For the presentation of the Principal Components Analysis (PCA) and Hierarchical Cluster Analysis (HCA), a Statistica 7.0 software was used.

RESULTS AND DISCUSSION

TSP and $PM_{2.5}$ Quantification

Montes Claros presents a central region which consists of narrow streets and in poor condition with a heavy flow of vehicles and individuals. Thus, the central areas of Montes Claros present environmental and infrastructure problems with special attention to the poor air and people circulation conditions. It also presents a significant industrial complex with targeted plants for the production of cement, fabric, drugs, bio-diesel and other products.

Table 2. Operating conditions of inductively coupled plasma mass spectrometer (ICP-MS).

Instrument	ELAN DRCII PERKIN-ELMER
Nebulizer	Meinhard type
Mist chamber	Cyclonic
torch injection	quartz (2mm)
RF Power	1100 Watts
Gas flow rate (L min $^{-1}$)	Nebulizer: from 0.56 to 0.98; Plasma: 15
Interface	Auxiliary: 1.2
Sampler	1.1 mm
Skimmer	0.9 mm
Replicates	3
Scans	40
Readings	1
Waiting time	50 ms
Voltage lens	6 V
Sample Introduction	1 mL min^{-1}
Correction equations:	Zinco: $^{64}Zn-(0.035297 \times {}^{60}Ni)$ Selenium: $^{82}Se-(1.007833 \times {}^{83}Kr)$

Table 3: Average concentrations of TSP and $PM_{2.5}$ in Central, Industrial and Control Areas in Montes Claros, Minas Gerais.

Area	Month	PTS/µg m^{-3} concentration	$PM_{2.5}$/µg m^{-3} concentration
Central	October	104.46	14.36
	November	85.57	14.46
	December	117.82	24.0
Industrial	October	48.15	7.91
	November	126.41	15.07
	December	139.95	12.18
Control	October	27.64	2.72
	November	18.97	5.38
	December	16.72	5.39

Despite the woodlands in the industrial area, there are gas emissions in various industries throughout the day, traces of unpaved streets and heavy flow of vehicles.

Viewing at data in the quantification of TSP and $PM_{2.5}$ as shown in Table 3, it can be noticed that the TSP concentrations encountered during the sampling period did not suffer considerable changes, except in October, in the industrial area, whose value is different as compared to other periods. For the central and control areas, the TSP values are similar.

In a paper developed in southern Brazil in industrialized urban areas, the weather and the topography of the region in relation to population distribution significantly contributes to the transport and dispersion of air pollutants. Thus, one can relate higher concentrations and smaller TSP and $PM_{2.5}$ with different influences on weather conditions (Carvalho and Jablonski, 2000).

The results showed that for the two regions prone to a higher incidence of particulate matter (central and industrial), the TSP levels meet the air quality standards in accordance with CONAMA 03/1990 Resolution. Figure 2 showed that sampling conducted in December in the industrial area had the highest amount of total particles.

CONAMA 03/1990 resolution is out of phase with the limits established by WHO and EPA and it has no standards for $PM_{2.5}$. It can be observed that Brazilian laws present standard for PM_{10} and the annual average for the established pattern is the least restriction (50 µg m^{-3}), while WHO recommends a lower value (20 µg m^{-3}).

By analyzing the data in Figure 3, it is observed that the central region sampled in December, has the highest concentration of $PM_{2.5}$, almost reaching the limit set by

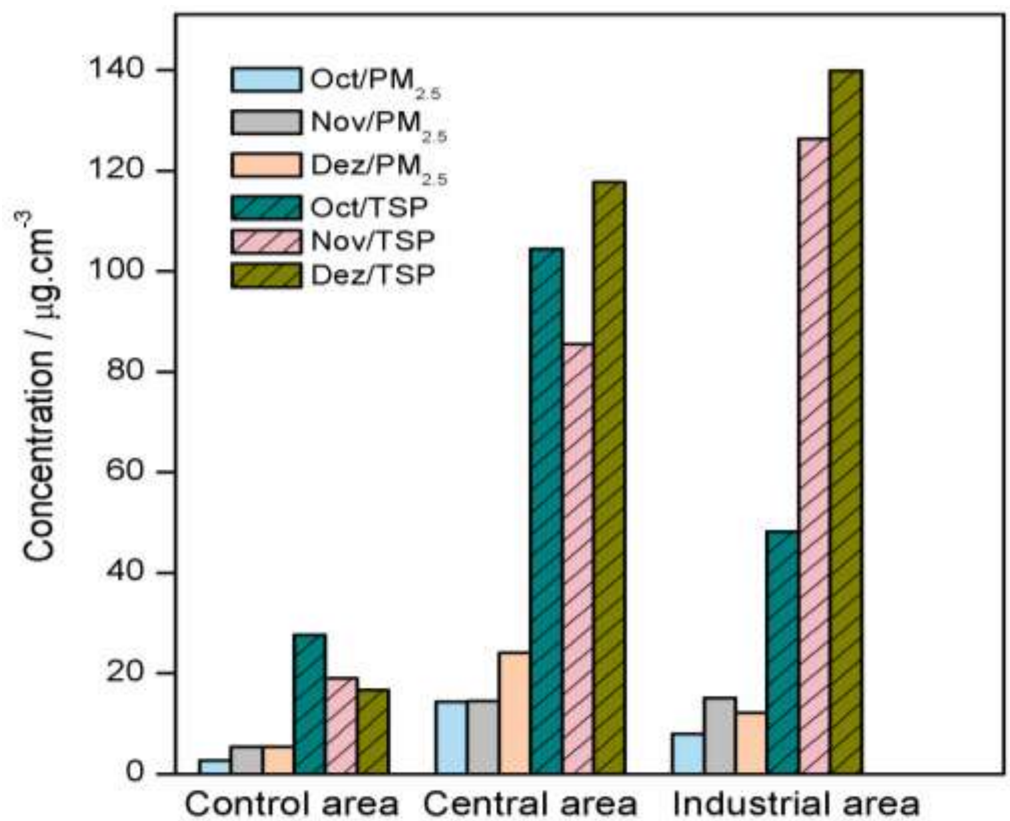

Figure 2: Descriptive results of TSP concentrations and $PM_{2.5}$ determined in different months of sampling for the control, central and industrial areas in Montes Claros, Minas Gerais.

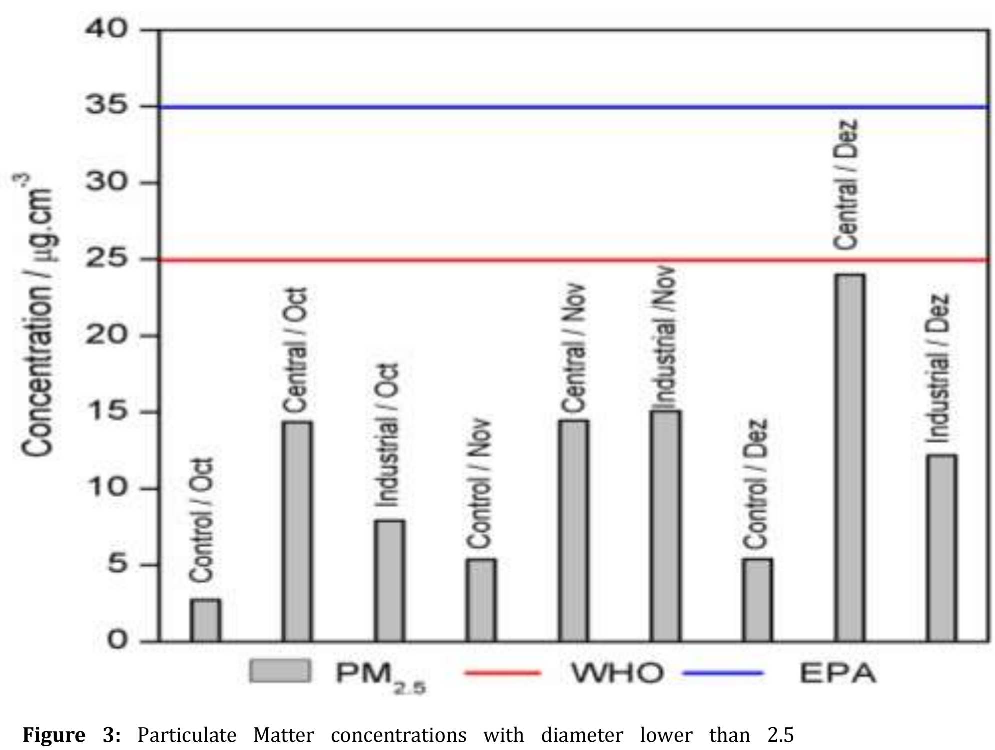

Figure 3: Particulate Matter concentrations with diameter lower than 2.5 micrometers in the three sampled areas (Control, Central and Industrial) in Montes Claros and WHO / EPA standards.

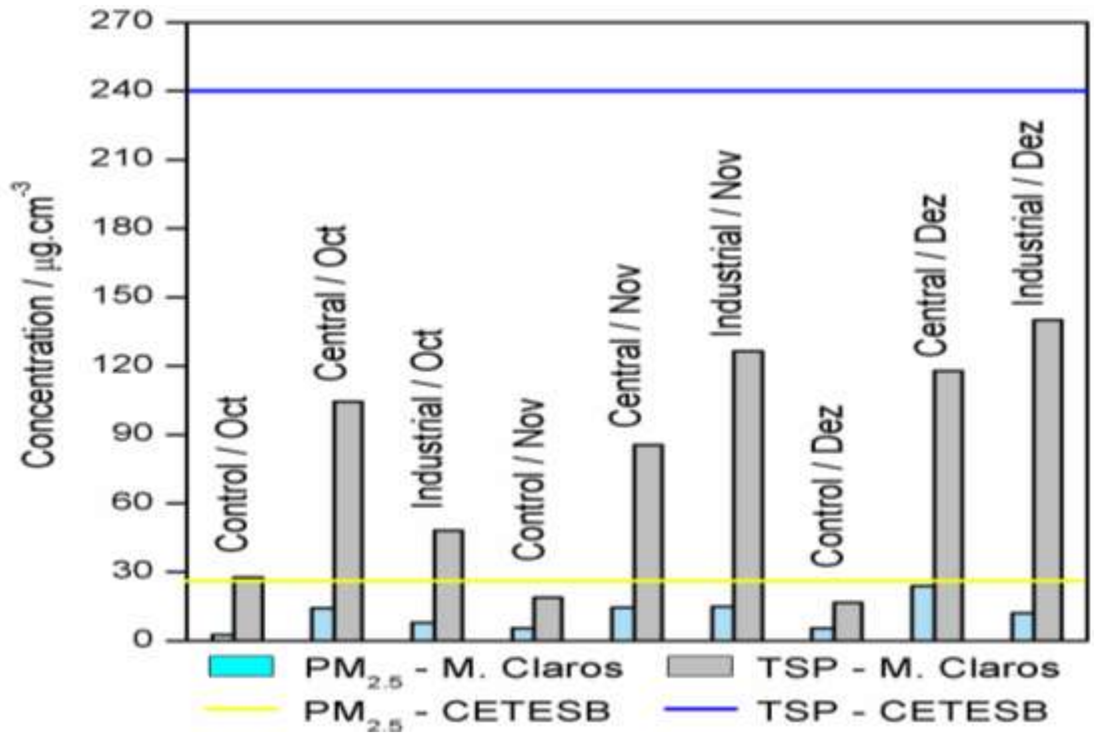

Figure 4: Concentrations of TSP in the three sampled areas (Control, Central and Industrial) in Montes Claros and indication of CETESB standards.

WHO for this parameter and weather conditions were not favorable to the removal of particulate matter, with wind speed considered null and without precipitation. For most regions and periods of sampling, concentrations remained below the reference standards. For sampling in the central region in October and November and industrial region in November, similar values can be noticed representing average values corresponding to 58.52 and 41.8% of WHO and EPA standards, respectively. Thus, one can relate higher concentrations and smaller TSP and $PM_{2,5}$ with different influences according to weather conditions.

In the days of collection of the central areas in November and the industrial areas in October, there was rainfall and the wind speed was 57 and 0.70 $m.s^{-1}$, respectively. In those days, the TSP and $PM_{2.5}$ concentrations were lower, signaling that there was performance of the wet removal mechanism.

The speed of the winds also interferes with the pollutant dispersion process by making them move provoking the decrease in that place, but the wind drag favors the emission and increases the concentration of pollutants in the atmosphere. Thus, in the days that larger TSP concentrations (139.95 $\mu g\ m^{-3}$) and $PM_{2.5}$ (24.0 $\mu g\ m^{-3}$) were found wind speeds were 0.0 and 1.22 $m.s^{-1}$.

For TSP and $PM_{2.5}$ values, the air quality standards monitored by the Environmental Company of the State of São Paulo (CETESB, 2014) were also considered as reference and they compared to the values found in this study (Figure 4).

The default value set by the $PM_{2.5}$ CETESB is 25 $\mu g\ m^{-3}$, That is, it is recommended by WHO. Regarding the TSP determined in Montes Claros and the standard CETESB, it is noticed that certain concentrations in the city are below the recommended limit.

Chemical characterization of the particulate matter

For chemical characterization of particulate material, the average concentration of the samples for each studied area was used. For a better interpretation of the results, these were submitted to the elements with the highest concentrations on the samples of $PM_{2.5}$ and TSP (Figures 5 and 6).

It was noticed that the chemical elements present in greater amounts in the TSP and $PM_{2.5}$ were Al, Ba and Zn in the central region, especially in October (Table 4). Some similarities were found between the concentrations of these elements in the month earlier mentioned in the central and industrial areas.

It is known that Zn comes mainly from the emission resulting from the combustion of vehicle fuels (Thorpe and Harrison, 2008). Its presence is associated with the use of additives and lubricants which have zinc compounds commonly added to engine oil by having anti-oxidant properties. The amount of this element on the determinations made in Montes Claros was significant and could be the elevated concentrations of Zn and Ba assigned

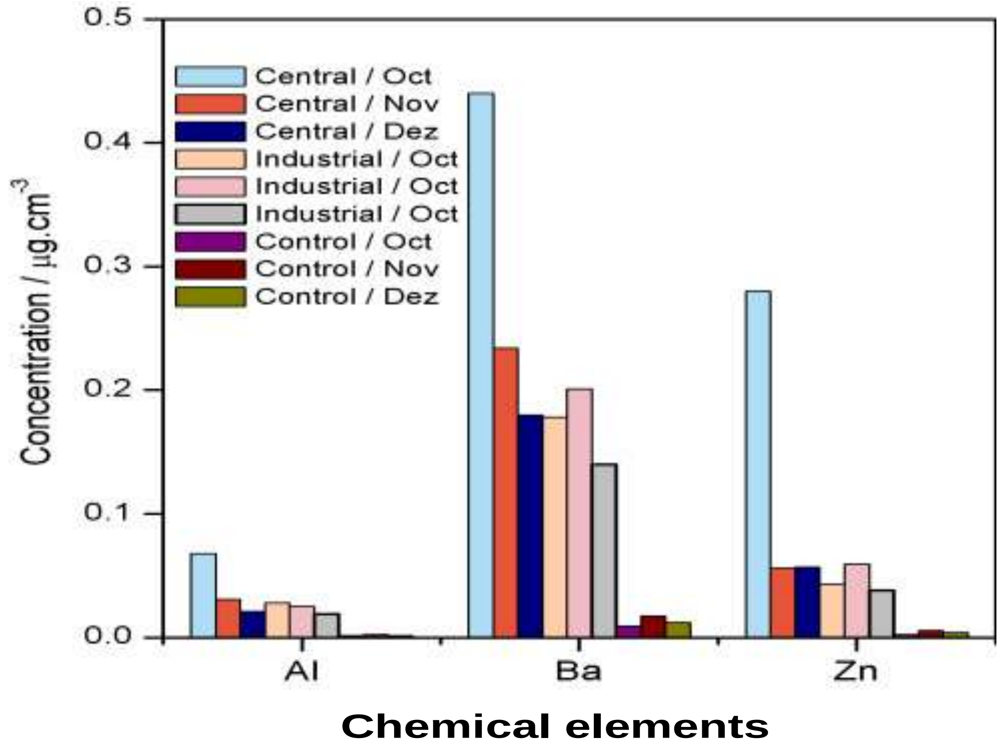

Figure 5: Concentrations of the chemical elements found in highest amounts in Particulate Matter samples with diameter less than 2.5 micrometers in different sampling areas (Control, Central and Industrial) in Montes Claros, according to different months (October, November, December).

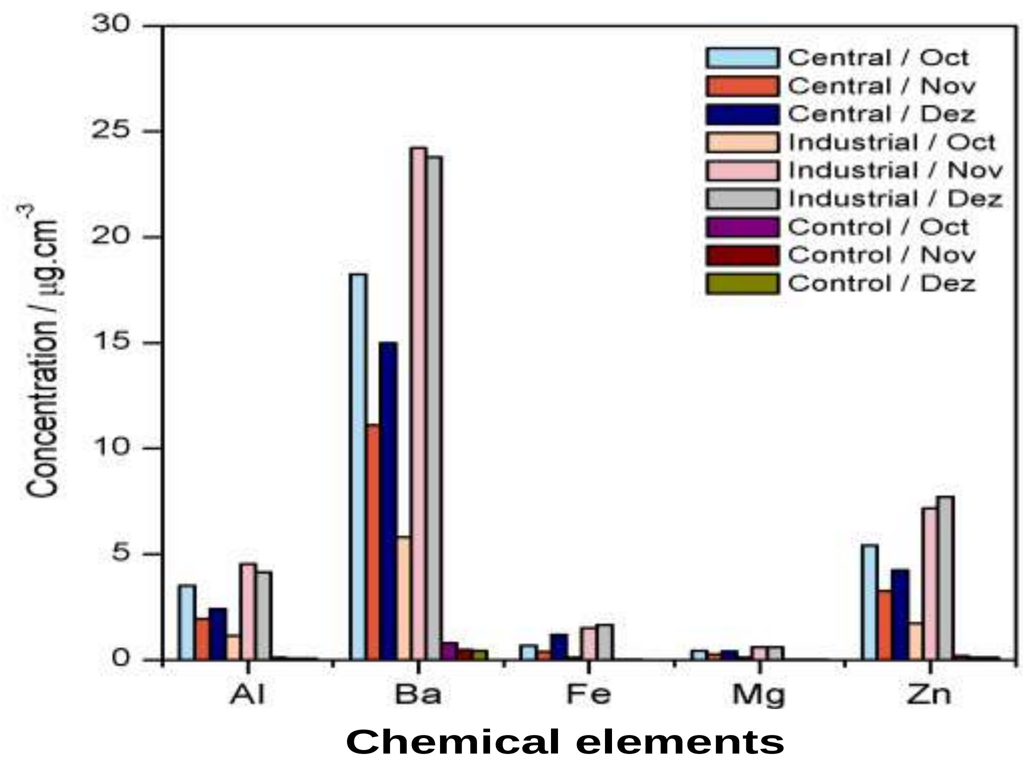

Figure 6: Concentrations of the chemical elements found in highest amounts in Total Suspended Particles in different sampling areas (Central, Industrial and Control) in Montes Claros, according to different months (October, November, December).

Table 4: Concentration of chemical elements (Zinc, Aluminum and Barium) were found in greater amounts in the central, industrial and control areas in Montes Claros in October, November and December, 2013.

Samples	**Concentrations/µg m^-3^**					
	TSP	**PM$_{2.5}$**	**TSP**	**PM$_{2.5}$**	**TSP**	**PM$_{2.5}$**
	Al		**Ba**		**Zn**	
Central/ October	3.507	0.068	18.245	0.440	5.419	0.280
Central/ November	1.947	0.031	11.116	0.234	3.268	0.056
Central/ December	2.409	0.021	14.999	0.180	4.247	0.057
Industrial/ October	1.131	0.028	5.805	0.178	1.734	0.043
Industrial/ November	4.538	0.025	24.221	0.201	7.169	0.059
Industrial/ December	4.138	0.019	23.798	0.140	7.713	0.038
Control/ October	0.108	0.001	0.794	0.009	0.189	0.002
Control/ November	0.059	0.002	0.477	0.018	0.126	0.006
Control/ December	0.058	0.002	0.440	0.012	0.103	0.004

Table 5: Reference values of chemical elements concentration in the air according to ATSDR.

Chemical element	**Concentration in the air/µg m^{-3}**
Aluminium	0.4 – 8.0
Barium	< 0.05
Cadmium	0,002 – 0.015 in urban areas; 0.015 – 0.15 for industrialized areas.
Chromium	< 0.020
Lead	< 0.050
Zinc	0.02 – 0,16 for urban areas.
Arsenic	0.02 – 0.1
Manganese	0.04
Selenium	0.01
Nickel	0.0022
Vanadium	0.011

to anthropogenic sources since these chemical elements are present in various components of the brake system in vehicles and also in fuels and lubricating oils.

The aluminum emission is probably related to the use of more modern catalysts that present substrates consisting of aluminum and magnesium silicates covered with aluminum oxide being released into the atmosphere abrasion (Thorpe and Harrison, 2008).

Table 5 shows some reference values of concentration of air chemical elements according to the Agency for Toxic Substances and Disease Registry (ATSDR, 2014) and comparing them with the results found in the chemical characterization of TSP and PM$_{2.5}$ in Montes Claros, lower concentrations of chemical elements such as Cadmium (Cd), Cobalt (Co), Copper (Cu), Manganese (Mn), Lead (Pb), Selenium (Se), Thallium (Tl), Vanadium (V), Arsenic (As) and Chromium (Cr) can be noticed.

As seen in Table 4 compared to the stipulated by ATSDR, it can be noticed that Ba concentrations are far above the limits, except for sampling of the control area with PM$_{2.5}$ (Table 6).

The Zn chemical element also exceeds the limits within results found for TSP in the central and industrial areas and PM$_{2.5}$, in the central area collected in October. Ni and V elements are also above the limits in the industrial area in the months of November and December for Vanadium and central area in December for Nickel (Ni). The element Aluminium (Al), although one of the most representatives of the higher concentration was not exceeded in relation to the limit indicated by ATSDR.

It is known that vehicles are considered potential source of metal emission through the wear pads and brake discs, rubber tires, gasoline and lubricating oil spill. The increase in the current vehicle fleet is among the factors of greatest pollution of anthropogenic origin. Several evaluations of vehicle emission rates made in tunnels in Brazil and other countries detected high concentrations of Cd, Pb, Cu and Zn (Grass et al., 2010) as well as, high levels of particulate

Table 6: Comparison between the concentrations of the chemical elements found in greater amounts in the air of Montes Claros in Central and Industrial area, and the limits established by ATSDR.

Chemical element	Area	Month	Concentration at TSP/ $\mu g\ m^{-3}$	Concentration at $PM_{2.5}$/ $\mu g\ m^{-3}$	Concentration in the air/ $\mu g\ m^{-3}$
Barium	Central	October	18.245	0.440	< 0.05
		November	11.116	0.234	
		December	14.999	0.180	
	Industrial	October	5.805	0.178	
		November	24.221	0.201	
		December	23.798	0.140	
Zinc	Central	October	5.419	0.280	0.02 – 0.16
		November	3.268	0.056	
		December	4.247	0.057	
	Industrial	October	1.734	0.043	
		November	7.169	0.059	
		December	7.713	0.038	
Nickel	Central	October	0.0011	8.39×10^{-6}	0.0022
		November	0.0007	3.63×10^{-6}	
		December	0.0013	6.92×10^{-6}	
	Industrial	October	0.0005	---	
		November	0.0020	---	
		December	0.0030	---	
Vanadium	Central	October	0.0066	9.24×10^{-5}	0.011
		November	0.005	9.36×10^{-5}	
		December	0.0108	1.28×10^{-4}	
	Industrial	October	0.0021	3.0×10^{-5}	
		November	0.013	1.1×10^{-4}	
		December	0.0171	7.0×10^{-5}	

matter and organic (Vasconcelos et al., 2005; Alves et al., 2014).

In a study in Abu Zaabal, Egypt, a high concentration of particulate matter containing Pb, Ni, Cr, Zn and Cd in the vicinity of industrial smelting areas was identified. This caused the contamination with toxic metals in nearby areas exceeding the limits established by the European Community. It was identified also that fertilizer plants account for a large amount of particulate matter contaminated by toxic metals (Ali et al., 2011).

The chemical elements Mn, Ni, Cu, Pb and Cr were found in low concentrations. The results suggest that the presence of these elements occurs due to impurities present in fuels and lubricating oils. Copper, for example, is added as an antioxidant and can be present in the coarser fraction of the particulate material (Silva et al., 2010). Their concentrations in the TSP sampling in Montes Claros presented the most significant result in relation to elements found in trace concentrations. This element is also emitted from the wear of bearings and other components related to the car's running engine. The use of copper as a reliable marker for the identification of brake wear particles in an urban environment is evident in some of the literatures (Thorpe and Harrison, 2008).

In order to obtain information on the sampled areas, chemical elements and its main correlations, the PCA and HCA were applied and presented in the form of dendrograms obtained for chemical elements in TSP and $PM_{2.5}$ (Figures 7 and 8).

The major similarities between the concentrations refer to the elements Zn and Al, Mg, Cu and Fe (Figure 7). The other groups of chemicals present very different concentrations being that the concentration of Barium (Ba) is the farthest from all others.

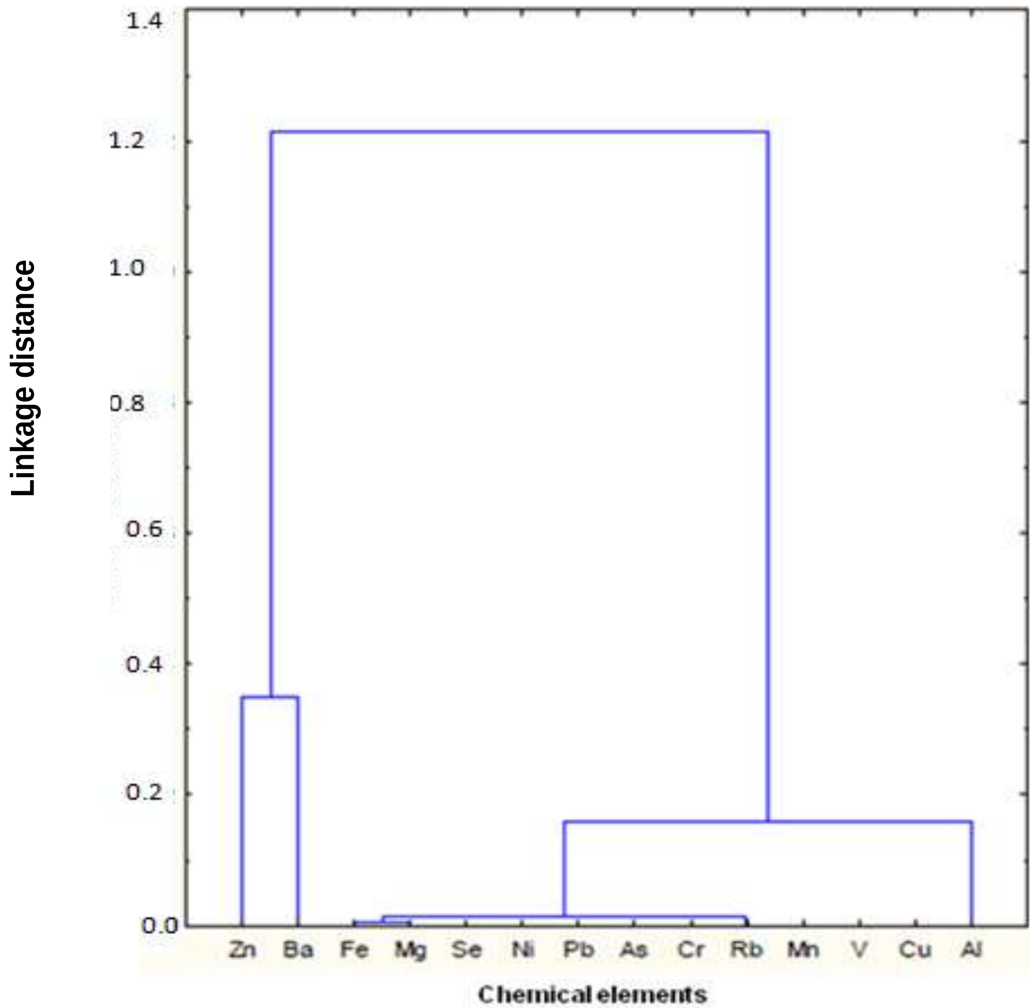

Figure 7: Dendrogram of relations between the concentrations of the different chemical elements found in the samples of TSP in the air of Montes Claros city.

Considering the $PM_{2.5}$, the major similarities among the concentrations of the chemical elements occur for Zn and Ba, Fe and Mg (Figure 8). The other chemical elements present very discreet similarities in their concentrations.

Figures 9 and 10 showed the results of the main components applied to the sampled areas in Montes Claros. The PCA 1 (factor 1) indicates that this component contains 79.47 and 88.28% of the information for the determinations of $PM_{2.5}$ and TSP, respectively. We can thus evaluate the similarity of samples and confirm that there was little loss of information described by the two components, that is, factors 1 and 2 (Borges, 2014).

It appears that the control area in all sampled months showed similarities with respect to the variables employed for TSP and $PM_{2.5}$. The sample of central / October $PM_{2.5}$ is the most different. Samples of industrial areas / December and industrial / October are similar, while the sample from the center / December is similar to the industrial / November, also for $PM_{2.5}$. For TSP, there were few similarities among the central and industrial areas, being that the similarity is discrete between center / December and industrial / November. Regarding the amount of particulate matter (139.95 μg m^{-3} TSP) and the concentration of chemical elements, the industrial area / December was considered as one with the biggest difference among the others.

Conclusions

According to CONAMA 03/90 resolution, the air quality in Montes Claros is classified as good in all sampled areas in the city. During the study period, no concentration of particulate matter was found, either PM2.5 or TSP, which reached the value according to important legislation of the primary standard for 24-h sampling.

The quantities of particulate matter presented differences during the study period due to weather conditions. Rainfall influenced their values, as there was significant variation in the days of little or no rainfall

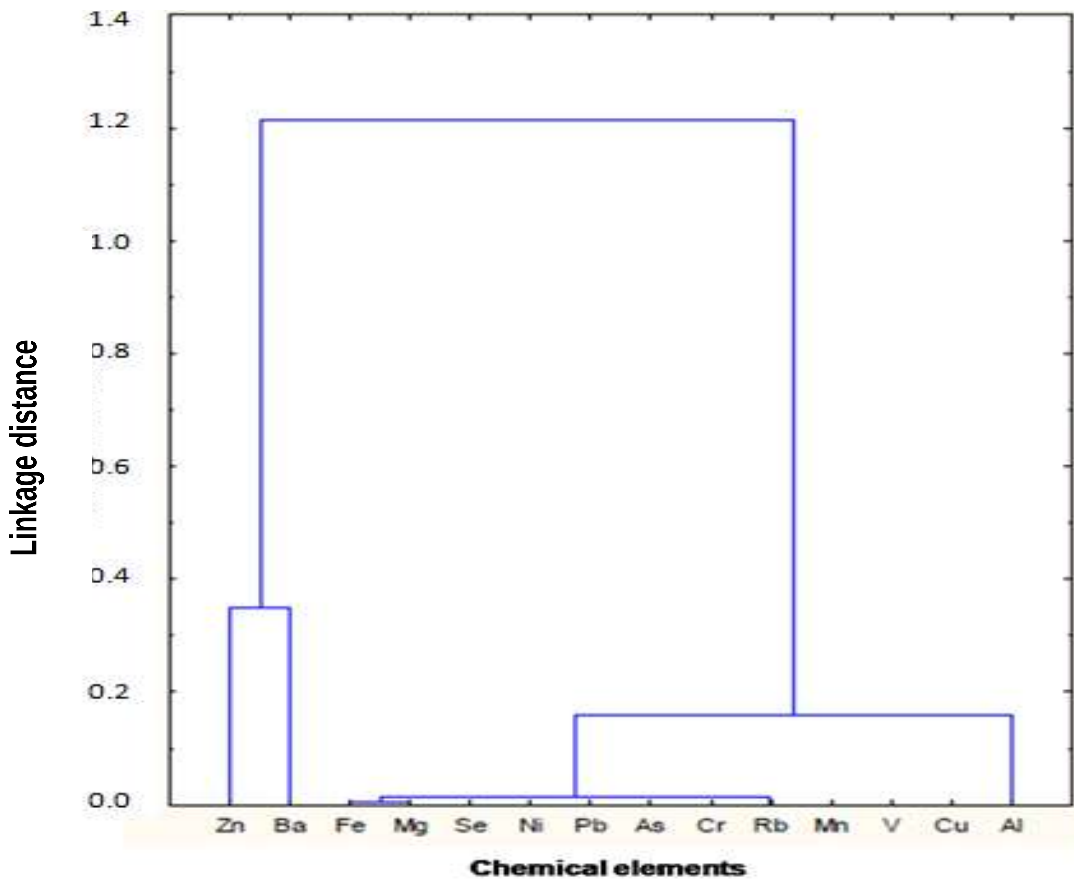

Figure 8: Dendrogram of relations between concentrations of different chemical elements found in Particulate Matter $PM_{2.5}$ in the air of Montes Claros.

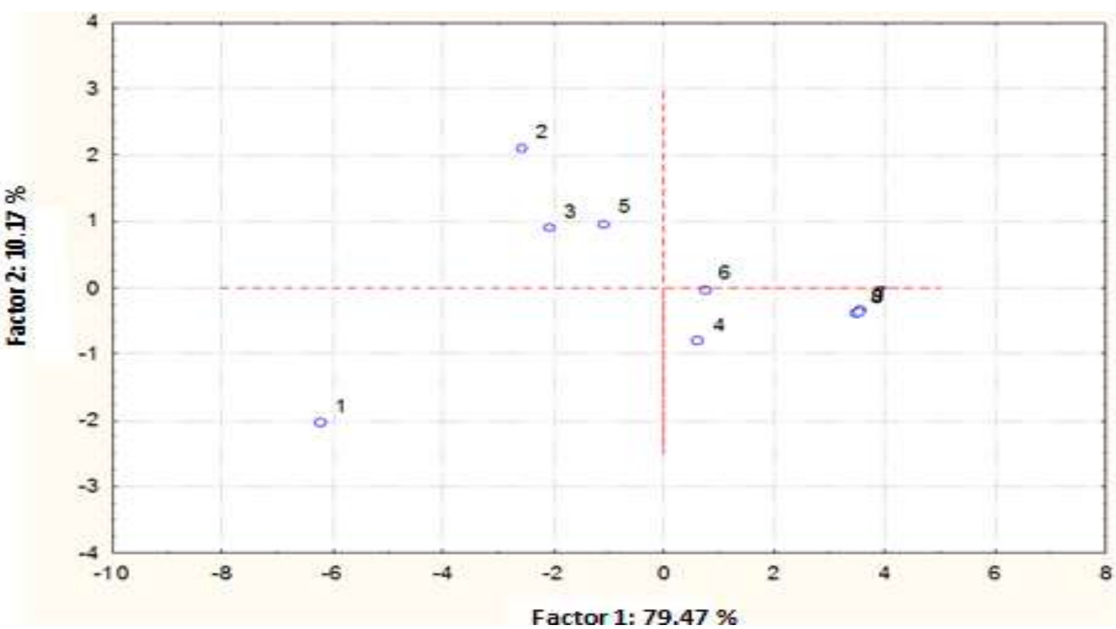

Figure 9: Analysis of Main Components applied to Particulate Matter with diameter less than 2.5 micrometers ($MP_{2.5}$) in the sampling areas (Central, Industrial and Control) in Montes Claros city. Legends: X - Central region/October; Y - Central region/November; Z - Central region/December; W - Industrial region/October; A - Industrial region/November; B - Industrial region/December; C - Control region/October; D - Control region/November; E - Control region/December.

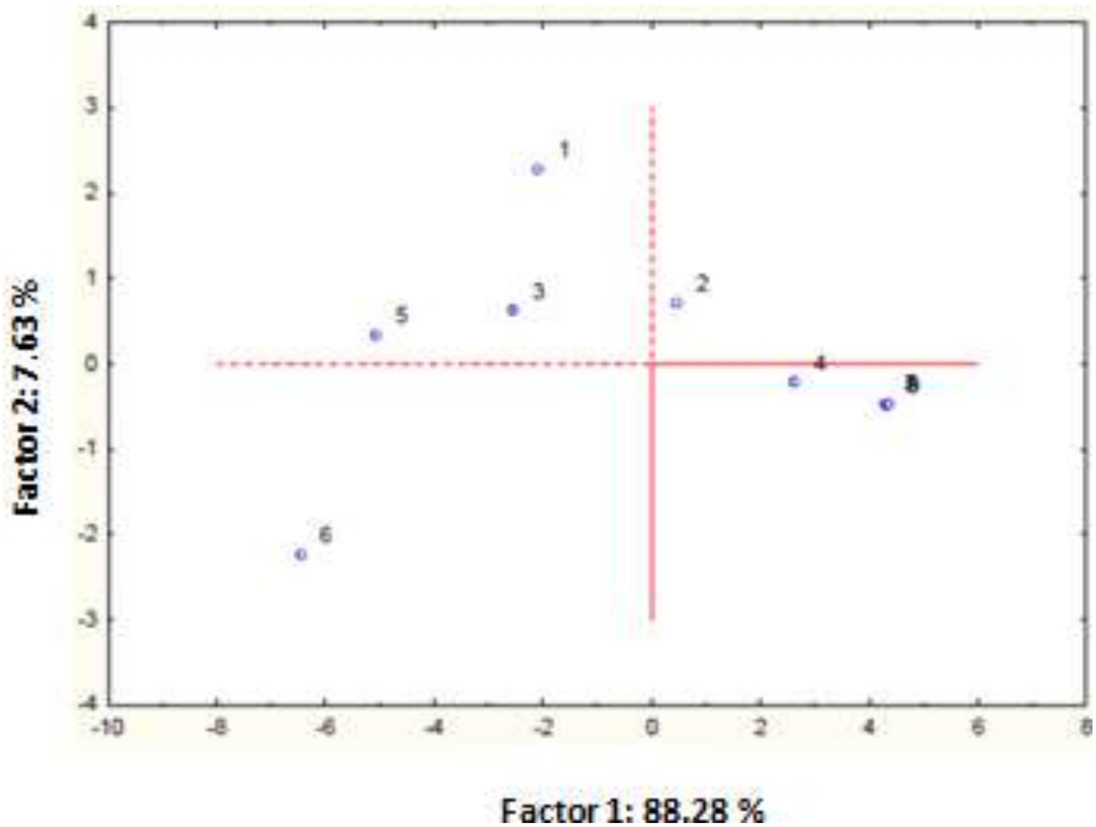

Figure 10: Analysis of the Main Components applied to TSP in the air of Montes Claros city in the different sampling areas (Central, Industrial and Control). Legends: X - Central region/October; Y - Central region/November; Z - Central region/December; W - Industrial region/October; A - Industrial region/November; B - Industrial region/December; C - Control region/October; D - Control region/November; E - Control region/December.

compared to most precipitation rainfall days in October (industrial area) and November (central area).

The concentrations of chemical elements present in the particulate matter showed variations for all the studied areas. However, Al, Ba and Zn described a very similar concentration profile in central and industrial areas with higher concentrations than the others.

The concentrations of Ba, Zn, Ni and V were above the limits recommended by legislation, highlighting the high concentrations of Ba and Zn in the central and industrial areas.

The sampled areas presented good correlation for the variables under study, highlighting the similarity of the control area, similarity between central and industrial areas in some months of sampling and most impacted places by vehicular flow indicating that this is the main source of particulate matter and chemical elements.

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