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Characterization Methods of Ions and Metals in Particulate Matter Pollutants on PM2.5 and PM10 Samples from Several Emission Sources

Mauricio A. Correa ¹, Santiago A. Franco¹, Luisa M. Gómez¹, David Aguiar¹ and Henry A. Colorado^{2,*}

- ¹ Grupo de Investigación y Laboratorio de Monitoreo Ambiental—GLIMA, Universidad de Antioquia UdeA, Calle 70 N°. 52-21, Medellin 050010, Colombia
- ² CCComposites Laboratory, Universidad de Antioquia UdeA, Calle 70 N°. 52-21, Medellín 050010, Colombia
 - * Correspondence: henry.colorado@udea.edu.co

Abstract: This research investigated the current methods of characterization of ions and metals in particulate matter pollutants from different emission sources. The study was conducted through the Proknow-C methodology, in which a portfolio that includes scientific and review articles was selected. The document addresses different methodologies currently used to quantify diverse ions and metals (IIMM) found in particulate matter (PM), specifically focused on PM_{10} and $PM_{2.5}$. The investigation was made going through the types of filters used to capture the pollutant, the equipment and the corresponding characterization techniques. Results show the Proknow-C method is a reliable way to analyze PM pollution research, revealing the state of art for metals and ions types, characterization technologies, current situations and trends. Sulfate, nitrate, and ammonium ions are found in concentrations between 70 and 80% of the PM. Among the main metals found are chromium, nickel, lead, cadmium, iron, manganese, coper, and zinc. The main detection method found in the studied research was inductively coupled plasma mass spectrometry. It was also found that geographic information systems are a good tool for integrating special data with PM and air pollution, which could accelerate the diagnosis and thus the actions to give solution to the problem.

Keywords: particulate matter; air pollution; trace elements; metals; ions

1. Introduction

Air pollution refers to the presence of harmful substances in the atmosphere in quantities that can be detrimental to human health and ecosystems [1–3]. It is related to emissions generated by industry [4,5], rapidly developing economic areas [6,7], vehicular emissions [8], and to adverse climatic conditions [9]. The main sources of pollution of human origin are waste burning in rural areas, mining and quarrying, manufacturing, chemical industry, oil refining, combustion engines and incinerators, urban transport, and energy production [10]. Pollutants actively intervene in the physics and chemistry of the atmosphere in processes such as cloud formation or radioactive balance [5,6,11], which also change the conditions of other environmental actors even altering the hydrological cycle [12]. Meteorological conditions such as climate [11], wind speed and direction [13], and precipitation levels [14], play an important role in the formation, transport, diffusion, and removal of atmospheric pollutants [2]. The atmosphere is one of the components of the environment most susceptible to the human activities [15].

Particulate matter (PM), also known as atmospheric aerosol or airborne dust [16], liquid or solid, which is a pollutant that contributes to environmental damage [17–19]. It directly influences the formation of haze [6,17,18,20] and ecological impacts [8], greenhouse effect, and climate change [8,12,17]. PM concentrations are used as an indicator of air quality [7]. PM can be transported through the air for extremely long distances [21] and stay suspended for long times [10]. It is a complex pollutant of not specific chemical and



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). physical composition [22,23], may consist on carbon compounds [12,24], ions [23,25,26], metals [27–29], hydrocarbons [30–32], among others [10,33,34]. Moreover, its composition may change over time according to the characteristics of the region [35]. PM is classified according its size into PM_{10} and $PM_{2.5}$ (aerosol which aerodynamic diameter is less than 10 µm and 2.5 µm, respectively) [27,36]. Depending on their origin, these can be classified as primary (direct emission of particles as such into the atmosphere) or secondary (generated in the atmosphere due to transformations of gases into particles) particles [37]. The characterization of these pollutants provides information about their chemical components, their origin, and emission sources. The composition of the PM_{10} and $PM_{2.5}$ samples will depend from the specific area where the sample is taken.

PM is widely investigated for negative environmental effects [38,39]. It's generated by several emission sources such as biomass burning [14], traffic [8], wildfires [40], industrial processes, and natural sources [41]. Wildfires contribute to particulate matter pollution worldwide, but more intensively in some areas such as California (USA) [42] and in the Amazonia region (mainly in Brazil, Perú, and Colombia). These fumes from wildfires have been shown to be toxic for lung macrophages [43], very critical for the human health because these cells surround and destroy microorganisms, eliminate dead cells, and stimulates other cells that contribute to the immune system.

PM is also investigated because it is a hazard for the human health [44–46]. These particles can be inhaled and enter the lungs causing harmful health effects [6], while finer particles may reach the alveolar region of the lungs [47,48]. The negative effects of PM₁₀ and PM_{2.5} depend on their complexity and variation of parameters, which include: particle size, heterogeneity, chemical composition, type of origin of the particle, pH, and solubility. Furthermore, depending on their concentration, exposure time, and toxicity, they can be lethal or cause major health problems in the short or long term. [16]. In addition, the mentioned characteristics may change over the time and with different weather conditions [41].

As an example of climatic conditions, in Medellin, Colombia, the geographical feature of mountains valley limits the dispersion of air pollution [14]. The unique shape of the valley, resembling a bowl, impedes the flow of wind and causes the accumulation of pollutants in the air. This phenomenon is especially pronounced during thermal inversion, when cold air from the surrounding mountains is held in place by a layer of warm air, creating a lid-like effect [4]. Figure 1a shows a typical emission source, while Figure 1b,c show two different city views with PM pollution at Medellin.



Figure 1. (a)Emission source and (b,c) Pollution in the city of Medellin.

Pollution in urban environments is one of the biggest challenges that exist today. PM negatively influences climate change, air quality, and consequently the health of human beings [49]. In 2016, about 4.2 million deaths worldwide were attributed to air pollution [50]. The main effects that PM causes on health are related not only to respiratory and cardio-vascular problems, but also to damage to multiple organs and tissues [35]. Due to the above, it is considered that this contaminant is linked to morbidities and premature deaths [28]. PM₁₀ is also known as the respirable fraction of PM, which is one of the main causes of health problems around the world due to the presence of toxic compounds in its composition, and to the ability to lodge in the lung tissues [51]. However, it has been

observed that finer particles ($PM_{2.5}$) can be more dangerous than PM_{10} since they can penetrate the alveolar region of the lungs [28]. Depending on their origin, these can be classified as primary (direct emission of particles as such into the atmosphere) or secondary (generated in the atmosphere due to transformations of gases into particles) particles [37].

PM is not the only environmental problem in Latin-America: the lack of appropriate regulations or law enforcements, and the lack of investment in circular economy aggravate the situation. However, some initiatives applied in Colombia and other countries are trying to change the trend of the region particularly in recycling solutions [52–54], education [55], and circular economy [56,57]. All these efforts could motivate the government to take difficult decisions to give solution to the PM contamination.

The goal of this article is to understand the current state regarding the characterization of ions and metals in PM_{10} and $PM_{2.5}$ samples. Some of the most common methodologies for sampling, analysis, and source detection for these metal species are included as well. The search keywords were carefully selected to find the characterization methods associated with metals and ions in particulate matter, also detailing PM_{10} and $PM_{2.5}$, and some of the most known technologies, but not limited to: AAS: Atomic absorption spectroscopy, ICP: induction plasma spectrometry, IC: ion chromatography, WSIS: Water-soluble ions. The filters and its associated technology are important to understand is limitations and advantages as well, now that pollution tests are more demanded worldwide, possibly requiring a new generation of them.

1.1. The Main Technologies

There are several important techniques used for the characterization of metals and ions in PM. Next, it is summarized the main technologies used for PM investigation.

Atomic Absorption Spectrometry is based on the light absorption by the atoms of the sample [58]. The amount of light that is absorbed by the atoms in the sample is measured, whose intensity is directly related to the concentration of the element in the sample. It is used to measure the concentration of elements in a sample. Ion Chromatography is a separation technique [59], based on the interaction of ions in a sample with a chromatographic column. The sample is injected onto the column and the components are separated based on their affinity for the column and their electrical charge. This technique is used to separate and identify components of a complex sample. Inductively coupled plasma mass spectroscopy is an ionization technique used to convert compounds into ions for further materials analysis [60]. A plasma is generated in a chamber and used to ionize the compounds in the sample. The generated ions can be analyzed with techniques such as mass spectrometry, chromatography, liquid chromatography, or X-ray diffraction for valuable information about the composition, structure, and properties of the materials [61]. Voltammetry is used to study the transfer of electrons in an electrochemical system. This is based on the measurement of the electrical current that flows through an electrochemical system in response to a change in electrical potential. It is used to determine the concentration of electroactive compounds in a solution and to characterize the electrochemical properties of materials. Liquid chromatography is a separation technique used to separate compounds in a solution [62]. The sample is injected into a column that contains an adsorbent material, where the compounds are separated on the column based on their affinity for the adsorbent material and their flow rate through the column. It is widely used for the analysis of complex compounds. X-ray diffraction is a characterization technique used to identify the crystalline structure of materials [63]. It allows the identification of complex crystalline structures and the detection of impurities in materials. It is a versatile technique and can be used to analyze a wide range of samples. Neutron activation is a technique used for the characterization of metals and their elemental composition [64], based on bombarding a metal sample with neutrons, which results in the formation of radioactive isotopes. These isotopes emit gamma radiation, which can be detected and analyzed to determine the chemical composition of the sample.

1.2. The Filters

The correct selection of the type of filter ensure the completeness and accuracy of results obtained in PM_{10} and $PM_{2.5}$ sampling. The main filters used are presented below, classified by their constitutive materials.

The fiberglass filter is a strong and durable, ideal for sampling in harsh environments [65]. However, it could release glass fiber particles into the sample during the filtration process, which can affect the integrity of the sample and lead to erroneous results in the subsequent analysis. Polyurethane foam is lightweight and inexpensive, commonly used elsewhere [66]. One limitation is that it can be quite porous and not retain all the particles in the sample, which can result in loss of information and inaccurate results. Quartz filter is inert and thus, it does not affect the composition of the sample [67], making it ideal for sampling sensitive chemicals. It could be more expensive than other filters, limiting their use in some regions. Teflon filter is resistant to high temperatures and corrosion, making it suitable for sampling in harsh environments [68]. However, it can also be more expensive when compared to others, and potentially contaminate the sample with Teflon particles during the filtration process. Aluminum filter is cheap and available elsewhere [69], but it can be sensitive to corrosion therefore reacting with certain components of the sample [70]. Polycarbonate filter is ductile and strong, making it suitable for sampling in environments durability is required. However, it can also be prone to damaging the integrity of the sample. Biofilters such as tree leaves are a natural and affordable material [71], but they are non-uniform materials affecting the effectiveness in the filtration process.

Therefore, the best filter for sampling PM_{10} and $PM_{2.5}$ depends on many factors, including the environment, costs, and measurement accuracy requirements. In general, teflon and quartz filters are considered the most suitable for sampling PM_{10} and $PM_{2.5}$ due to their inertness and resistance to corrosion, which reduces the possibility of sample contamination. However, it is recommended to conduct a detailed evaluation depending on the specific filter application.

2. Methods

The instrument of intervention Knowledge Development Process—Constructivist (ProKnow-C), was used in this research to undertake an analysis of the characteristics of the publications. The ProKnow-C was developed from 1994 by the Laboratory of Constructivist Decision Aid Methodologies (LabMCDA-C), of the Federal University of Santa Catarina [72]. The methodology is divided into two steps: data collection and information analysis:

2.1. Data Collection

The development of this step is about selecting a representative fragment of the literature relative to the "Characterization of metal and ions in PM_{10} and $PM_{2.5}$ ", addressed both in theoretical and empirical research. Thus, for conducting this research, the methodology used is based on a structured selection and analysis of literature of the area, aiming the construction of knowledge on a particular subject, following a constructivist view. This allows a critical analysis of the bibliographic portfolio (BP). The selection of the BP was performed searching the commands of the Table 1 in the international databases of research, commands selected after analyze target questions, keywords, and thesaurus.

 Table 1. Searching commands selected through the Proknow-C methodology.

Туре	Search Command
1	(Characterization or analysis) and (PM_{10} or $PM_{2.5}$) and (metals or ions)
2	(Characterization or analysis) and (PM_{10} or $PM_{2.5}$) and (AAS or ICP)
3	(Characterization or analysis) and (PM_{10} or $PM_{2.5}$) and (IC or chromatography)
4	(Characterization or analysis) and (PM ₁₀ or PM _{2.5}) and (Metals or trace elements)
5	(Characterization or analysis) and (PM_{10} or $PM_{2.5}$) and ((anion or cation) or WSIS)

AAS: Atomic absorption spectroscopy, ICP: induction plasma spectrometry, IC: ion chromatography, WSIS: Water-soluble ions.

The procedure to obtain the final portfolio of documents begins with the target questions that allow summarizing the subject to be investigated. Subsequently, keywords and boolean operators were selected to build search commands, the databases in which the information will be searched are selected and the delimitations are established as needed. When searching for the commands in the databases, an initial portfolio of 3542 documents (scientific articles, review articles, and books) were obtained. Using a bibliographic manager such as Mendeley, the repeated documents were eliminated, while a second portfolio of 1121 was obtained, from which the relevant information is selected by its abstract and/or title. After this step an amount of 103 documents obtained, and deeply analyzed. Finally, it was found that the most important articles are 41, being the center of the analysis. The processes made in the step one is resumed in the Figure 2.



Figure 2. Summary of the process to get the bibliographic portfolio.

2.2. Information Analysis

After the selection of the articles which composed the BP, they were analyzed in 4 main points: (I) Identification of most representative articles. (II) Principal instruments of sampling, to analyze the material of the filters, the devices used to catch the pollutant, and the different kind of methodologies carried out to get the samples. (III) Characterization of IIMM, that include all the different techniques used to measure the concentration of IIMM, the conservation and preparation of the samples. And (IV), the main species found, which are the main IIMM in PM_{10} and $PM_{2.5}$ samples, which are the most characterized.

3. Results

After selecting the documents of interest through the methodology, a total of 41 relevant articles were chosen to be analyzed in the bibliographic portfolio, later used as the basic references in the analysis. It was found that there is a wide variety of filter materials to collect samples of PM_{10} and $PM_{2.5}$ pollutants. These include quartz [73], fiberglass, Teflon, aluminum, polyurethane, and even studies that have used biofilters, such as Malaleuca leaves [51]. Even special bags are used for pollutant collection [74]. There is no specific differentiation for filter selection according to the pollutant to be collected and measured, nor is there a differentiation in the choice of filter are used to perform characterization of ions and metals. Regarding the collection equipment, high, medium and low volume devices were found. There are even devices that perform the characterization of pollu-

tants automatically, such as optical counters [75]. Also, acid digestion is used to extract contaminants from the filters.

A total of 41 metals were found in these papers: Aluminum (Al), Barium (Ba), Beryllium (Be), Cadmium (Cd), Calcium (Ca), Cerium (Ce), Cesium (Cs), Chromium (Cr), Cobalt (Co), Copper (Cu), Europium (Eu), Iron (Fe), Gallium (Ga), Gold (Au), Hafnium (Hf), Mercury (Hg), Magnesium (Mg), Manganese (Mn), Neodymium (Nd), Nickel (Ni), Potassium (K), Lead (Pb), Samarium (Sm), Scandium (Sc), Sodium (Na), Silver (Ag), Strontium (Sr), Tantalum (Ta), Terbium (Tb), Thallium (Tl), Thorium (Th), Tin (Sn), Titanium (Ti), Tungsten (W), Uranium (U), Vanadium (V), Ytterbium (Yb), Zinc (Zn), and Zirconium (Zr). On the other hand, 11 ions were characterized: Calcium (Ca⁺²), Magnesium (Mg⁺²), Sodium (Na⁺), Potassium (K⁺), Ammonium (NH4⁺), Chloride (Cl⁻), Sulfate (SO4⁻²), Nitrate (NO3⁻), Nitrite (NO2⁻), Fluoride (F⁻), and Phosphate (PO4⁻³)

Figure 3a shows the number of papers of the BP that contains analysis by the type of filter, where the quartz filter stands out, followed by the glass fiber. Similar to Figure 3a, the analysis of the literature with respect to the characterization techniques is performed in Figure 3b, highlighting ion chromatography as the main technique for the characterization of ions. On the other hand, for the analysis of metals mass spectrometry and atomic absorption spectroscopy are the main ones. In Figure 3c, the most relevant metals for research (lead, cadmium, nickel, chromium and copper are the most studied) are shown; while in Figure 3d, the main ions are shown, highlighting the chloride, nitrate and sulfate anions; and the sodium and ammonium cations.



Figure 3. (a) Type of filter, (b) Characterization technique, (c) Main metals characterized, (d) Main ions characterized vs. number of documents.

Figure 4a shows the number of papers found in the search classified by the species, as metals (28), ions (26) and both species (13). Figure 4b shows the number of documents involving PM_{10} (27), $PM_{2.5}$ (38), and both (24).



Figure 4. Venn diagram for (a) Analyzed substance and (b) Pollutant type vs. number of documents.

3.1. Filters Types and Parameters

It was found that quartz filters were the most used, followed by fiberglass, polyurethane, Teflon and aluminum filters. Other capture methods were analyzed as well, such as Tedlar bags or biofilters (such as Malaleuca leaves). Some documents are not specific with the type of filter used. Filters of different porosities were used, the preferred one being the 47 mm Whatman filter. It was determined that quartz, glass fiber, polyurethane, and Teflon filters, all used to capture PM_{10} and $PM_{2.5}$ samples and to characterize both ions and metals. Table 2 shows the analysis of the physical characteristics of the filters.

 Table 2. Filter physical characteristics.

Filter	Pore	Brand	M or I	Pollut	ant	Reference
	47 mm	Whatman	Metals	PM ₁₀	PM _{2.5}	[35]
-	47 mm	Whatman	Metals	PM ₁₀	-	[76]
Fiber glass	47 mm	Whatman	Ion	PM ₁₀	-	[25]
-	-	Whatman	-	PM ₁₀	-	[35]
DUE (Delyurathana feam)			Metals	DM	DM ([00]
FOF (Folyurethane loam)	-		Ion	– Pivi ₁₀	PM _{2.5}	[28]
	417		Metals	_	PM _{2.5}	[00]
	47 mm	Palifiex -	Ion			[29]
-	150 mm	Sartorious	Metals	PM ₁₀	-	[77]
-	47 mm	Whatman	Ion	-	PM _{2.5}	[78]
-	47 mm	-	Ion	PM ₁₀	PM _{2.5}	[26]
Over the Class	47 mm	XA71 (Ion	DM	-	- [79]
Quartz fiber		Whatman	Metals	$ \Gamma NI_{10}$		
-	17	X471 /	Ion		PM _{2.5}	
	47 mm	Whatman –	Metals	_ •		
-	-	-	-	PM ₁₀	PM _{2.5}	[80]
-	-	Tissuequartz	Ion	PM ₁₀	PM _{2.5}	[23]
	-	-	Metals	PM ₁₀	-	[81]
Teflon	47 mm	Whatman	Ion	PM ₁₀	-	[11]
1011	37 mm	-	Metals	PM ₁₀	PM _{2.5}	[82]

Filter	Pore	Brand	M or I	Pollut	ant	Reference
Alumium	25 mm	-	Metals	-	PM _{2.5}	[21]
Tedlar bag	_	-	Metals	_	PM _{2.5}	[74]
Leaves	-	Malaleuca	Metals	-	PM _{2.5}	[51]
Polycarbonate	-	Essque	Metals	PM_{10}	PM _{2.5}	[83]
-	-	-	Ion	-	PM _{2.5}	[84]
	37 mm	-	Metals	PM ₁₀	PM _{2.5}	[85]

Table 2. Cont.

Among the measuring devices, high-volume and low-volume collection samples of PM were found to be the preferred for researchers. Table 3 shows the characteristics of the collection devices according to the filter used. the pollutant extraction technique is also shown, where acidification with nitric acid (HNO₃) and microwave extraction stand out.

Table 3. Kind of sampling device and pollutant extraction according the filter.

T !!/		Measuring Device		D (
Filter	Kind	Model	Brand	Pollutant Extraction	Keterence	
	× 1	LVS3.1			[27]	
	Low volume	PNS16T-3.1	-	Microwave extraction		
Fiber glass	High volume	460NL	Envirotech	Acidification with HNO ₃	[76]	
	High volume	APM 460 BL	Envirotech	Ultrasonication	[25]	
	Respirable dust	460NL	Envirotech	Acidification with HNO ₃	[35]	
PUF (Polyurethane foam)	MicroDust Pro Real	HB3275-07	Casella	method IO-3.1, US-EPA	[28]	
	High volume	KB1000	Jinshida	-	[29]	
	High volume	-	MCV	Acidification with HNO ₃	[77]	
	Four channel sampler	TH-16A	Tisnhong	-	[78]	
Qartz fiber	Mini-Vol portable	-	Airmetrics	Ultra pure-water	[26]	
	High volume	-	Andersen Sierra	Sonication and multiwave	[79]	
	Low volume	PQ200	BGI	extraction		
	High volume	GMB2360	Thermo		[80]	
	Low volume	2025	Partisol	- (USMW)		
	High volume	-	Tish	Ultrasonication	[23]	
	_	-	Anderson	Acidification with HNO ₃	[81]	
	Med volume	TH-150F	Tianhong	-	[5]	
Teflon	Four channel sampler	TH-16A	Tianhong	Ultrasonic agitation	[78]	
	Dichotomous	241	Anderson	Acidification with HNO ₃	[82]	
Alumium	Low P. impactor	DLPI	Dekati	Acidification with HNO ₃	[21]	
Tedlar bag	- EPAM 5000		Hazdust	Acidification with HNO ₃	[74]	
Leaves	-	-	-	Acidification with HNO ₃	[68]	
	Automatic	MARGA	MARGA	-	[84]	
-	Dichotomous	241	-	Acidification with HNO ₃	[85]	

3.2. Characterization Techniques

The methodologies for the characterization of IIMM range from the way in which the sample is taken, the collection de-vice, the characterization technique, and other parameters. Glass fiber, quartz, cellulose membrane, biofilters, and other filters can be used. The collection devices can be Hi-Vol, Med-Vol, Low-Vol, impactor, automatic, etc. As for the material extraction techniques, acid digestion is usually employed, although other alternatives exist, such as microwave extraction. Species analysis techniques are differentiated, where ion chromatography (IC) is commonly used to characterize ions, while atomic absorption spectroscopy (AAS) and induction plasma spectrometry (ICP) are used for metals.

Table 4 shows the characterization techniques used to analyze the chemical components of the filters of the study. For metals, the most commonly used techniques are AAS and ICP, while for ions IC is typically used. Table 4 also shows the methods used to characterize the samples, and the methodologies used to determine the emission sources, where principal component analysis and factorization matrices stand out. Finally, the chemical components found, the pollutant analyzed (PM_{10} or $PM_{2.5}$), and the sampling period are described as well. Other methods to characterize metals found were Inductively coupled plasma atomic emission spectroscopy (ICP-AES), Inductively coupled plasma—optical emission spectrometry (ICP-OES), X-ray absorption spectroscopy (XAS), X-ray diffraction (XRD) and Neutron activation analysis (NAA). For ions, there are high performance liquid chromatography (HPL) and Ultraviolet–visible spectroscopy (UV-VIS).

Technique	Device	Followed Method	Sources Detection	Chemical Compound	Pollutant	Sampling Period	Ref.
	ZEEnit 700— Analytic Jena GmbH	EN12341	-	Al, Mn, Fe, Ni, Cu, Zn, Sn, Pb, Si, Mg, Cr, As, Na, K, Ca, Sr, Cd	Both	December 2017–November 2018	[27]
	Thermo AA, Solar-Series	Method IO-3.2, US-EPA	-	Pb, Cd, Ni, Zn, Fe, Cu, Mg, Na, Ca, K	Both	June 2012–April 2013	[28]
	GBC-Avanta	-	USEPA PMF5.0	As	PM ₁₀	April 2010–December 2011	[76]
	SOLAAR 969	-	-	Ca, K, Mg, Na, Al, Pb, Cr, Ni, Zn, Cu, Cd, and Fe	Both	December 2009–December 2010	[85]
	GBC, Avanta, Australia	-	-	Fe, Cu, Zn, Mn, Cr, Cd, Pb, Ni	PM ₁₀	December 2008–January 2009	[35]
IC	IC Vario-940 Metrohom, Switzerland			Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻ ,	Both	June 2012–April 2013	[28]
	-	-	-	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻ ,	PM _{2.5}	-	[84]
	881 Compact IC Pro, Switzerland	-	-	Ca ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻ , F ⁻	PM _{2.5}	November 2015–April 2016	[29]

Table 4. Characterization techniques and its characteristics.

Technique	Device	Followed Method	Sources Detection	Chemical Compound	Pollutant	Sampling Period	Ref.
	Thermo Dionex; Sunnyvale, CA	US EPA protocols (Chow and Watson, 1998)	-	$\begin{array}{c} Na^{+}, NH_{4}^{+}, \\ Cl^{-}, SO_{4}^{-2}, \\ NO_{3}^{-}, \\ NO_{2}^{-}, F^{-}, \\ PO_{4}^{-3} \end{array}$	Both	January 2018–October 2018	[26]
	AS12A, ASRS Ultra II CD20 Dionex	-	-	Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻	Both	March and April 2009	[79]
	Dionex QIC CS12A	-	-	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺	Both	March and April 2009	[79]
	Thermo Fisher Scientific Inc., USA	-	USEPA PMF5.0	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻	Both	June and December 2018	[5]
	Dionex	-	-	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻	Both	December 2006–January 2007	[23]
	Metrohm 882 Professional IC	-	-	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻	PM ₁₀	December 2010–October 2014	[25]
	-	EN-UNE 14902	-	V, Mn, Fe, Ni, Cu, Zn, As, Mo, Cd, Sb, Pb	PM ₁₀	January 2015–January 2016	[77]
	ICP-MS 7700× system (Agilent Technologies, USA)	-	PMF: Paatero and Tapper (1994)	Ag, Al, As, B, Ba, Be, Bi, Cd, Co, Cu, Cr, Fe, Ga, Mg, Mn, Mo, Ni, Pb, Rb, Se, Sr, Te, Ti, U, V, Zn	PM _{2.5}	2016	[21]
	-	Compendium Method IO 3.5	-	As, Be, Cd, Cr, Co, Pb, Mn, Ni	PM ₁₀	July 2017	[81]
ICP-MS	ICP-MS, Agilent Technologies, model 7500 CE	-	-	As, Cd, Cr, Cu, Mn, Ni, Pb, Sb, V	Both	-	[82]
	NexION-350, Perkin Elmer, Inc., USA	-	-	Pb, Ni, Cr, As, Cd, V	PM _{2.5}	-	[74]
	Thermo X2 Series	-		Al, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Sr, Cd, Ba, Pb	PM _{2.5}	November 2015–April 2016	[29]
	Thermo Finnigan, Bremen, Germany	-	USEPA PMF5.0	Al, Ca, Fe, Mg, Zn, Ba, Cd, Cr, Cu, Mn, Pb, As, Ni, Se	Both	June and December 2018	[5]

Table 4. Cont.

Technique	Device	Followed Method	Sources Detection	Chemical Compound	Pollutant	Sampling Period	Ref.
Voltametry	-	-	USEPA PMF5.0	Pb, Cu, Cd	PM ₁₀	April 2010–December 2011	[76]
ICP-AES	Jobin Yvon, Model ULTIMA 2	-	USEPA PMF5.0	Al, Mn, Cr, V, Fe, Ni, Zn	PM ₁₀	April 2010–December 2011	[76]
ICP-OES	iCAP 6500 (Thermo Scientific)			Ca, Al	Both	March and April 2009	[79]
ICP-OES	ICP-OES, Termo Scientifc iCAP 7000 series	-	-	Al, Fe, Zn, Pb, Cu, Mg, Co, Ba Cr K Ca Mn	PM _{2.5}	October 2019	[51]
	iCAP 6000 Series (Thermo Scientific, Mas- sachusetts, USA)	-	-	Al, As, Ca, Cd, Co, Cr, Cu, Dy, Er, Eu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, Pr, Sn, Tb, Ti, V, Zn	Both	October 2018 and January 2019	[80]
LC	LC20AD, Shimadzu Corp., Kyoto, Japan	-	-	Ca ⁺² , Mg ⁺² , Na ⁺ , K ⁺ , NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻	Both	January 2013	[78]
HPLC	WATERS IC-pak TM			NH4 ⁺ , Cl ⁻ , SO4 ⁻² , NO3 ⁻	PM ₁₀	June 2004–February 2005	[86]
XAS	MRCAT			As, Pb	PM ₁₀	-	[36]
SUV-VIS	HACH DR 5000			NH ₄ ⁺ , Cl ⁻ , SO ₄ ⁻² , NO ₃ ⁻ , NO ₂ ⁻ , PO ₄ ⁻³	PM ₁₀	-	[11]
NAA	-	-	-	Ba, Br, Co, Sb, Cr, Fe, Ca and Zn	PM ₁₀	-	[87]
XRD		-	-	Fe As	PM ₁₀	-	[88]

Table 4. Cont.

4. Analysis and Discussion

PM is mainly composed of inorganic ions, mineral matter, and carbon compounds [75]. These compounds occupy between 40% to 50% of the mass composition in PM_{2.5}. Metals occupy between 1% and 5%, and are subject of high scientific interest because they are highly related to the morbidities associated to PM [23]. The presence of metals in PM is mainly due to anthropogenic activities such as industrial production [89], vehicular emissions [76,77,89], or other origins [87,89]. Metallurgy is a source of metals such as Mn, Ni, Zn, Cr, Fe, Cu, Cd and Pb [90]; while construction industry is responsible for others such as Al, Cu, K Mg, and Ca. In places where large amount of Ni is found, more respiratory and cardiovascular diseases are reported [90]. The exposure to these metals can be dangerous even in small concentrations [83].

Among the selected articles, almost only ions that were soluble in water (WSIS) were found to be characterized, which is because they are the most relevant in the composition of PM [78]. The most studied ions are sulfate (SO_4^{-2}), nitrate (NO_3^{-1}) and ammonium (NH_4^+), likewise, the fraction of these ions occupy between 70–80% of the total concentration among the IS present in the PM [85]. Other important ions are: chloride (Cl^-), phosphate ($PO_4^{3^-}$)

and fluoride (F^-) and the cations: calcium II (Ca^{+2}), potassium (K^+), sodium (Na^+), and magnesium II (Mg^{+2}).

There are different methodologies to measure PM emission sources. One of the outstanding one is to develop the positive matrix factorization algorithm (PMF) [28]. The United States protection agency (EPA) has established protocols that are used to adequately execute this methodology, see the EPA PMF 5.0 (Table 4 shows which articles use the method). Another way to determine the emission sources consist of the analysis of geographic information systems, mainly satellite images [78]. The chemical mass balance analysis (CMB) is used as well, a method that focuses on direct emission points, which does allow to determine the emission sources of secondary pollutants because they are formed in chemical processes that occur in the atmosphere [5]. Regardless of how emission sources are defined, the results highlight natural sources (suspension of crustal material, sea salts, volcanic eruptions, among others) and anthropogenic sources (automobile, industrial, agricultural, biomass burning, among others).

Regarding the emission sources, the nitrate, sulfate and ammonium ions come from their precursor gases (NOx, SO₂, NH₃) that are usually emitted in the combustion of coal, industrial and agricultural processes, the high concentrations of these ions in the PM directly affect the degradation of the visibility of the atmosphere [78].

Normally, sulfate occupies the largest fraction among IS [22]. Cl^- and Na⁺ come mainly from sea salts [29], although chlorine can also come from coal combustion, home heating, and industrial processes, such as steel alloying [77]. Calcium ions come from the suspension of natural dust or due to agricultural and construction activities. The concentrations of calcium are regularly related to those of magnesium [29]. F^- is normally low in terms of concentration, among its emission sources are coal combustion processes and brick manufacturing [27].

The metals that covered the most relevance in the studies investigated were: chromium (Cr), nickel (Ni), lead (Pb), cadmium (Cd), iron (Fe), manganese (Mn), copper (Cu), and zinc (Zn), among others. In most studies the metal with the largest fraction was iron. The fraction of metals in PM is usually low, found at a trace level in PM samples. Figure 3c lists the metals that were mostly characterized among the selected studies.

Thallium (Tl) is a toxic heavy metal, that event at low concentration is more hazardous to mammals than Pb, Hg or Cd [91]. One of the human-generated sources of Tl is the sulphide ores from the extraction process [92,93]. Beryllium can be found in the atmosphere as a result of soil particles being lifted and dispersed from typically open mines or areas near factories that process raw materials [94]. Sn compounds are present in limited amounts in the Earth's surface and the appearance of tin in the atmosphere is largely due to human activities, such as burning fossil fuels, and industrial and refining processes [95]. Besides, tin comes from the burning of fossil fuel and waste incineration [96] and other metals comes from glass making, electronics, and mechanical industries [97]. Titanium oxide (TiO₂) serves as a material that can withstand high temperatures therefore used in various industries [98]. Also, crushed titanium (Ti) is increasingly being used in the aerospace, automotive, and other fields for its light weight and strength properties [99]. Powder form of titanium carbide (TiC) is used in the manufacture of strong alloy tools, carbide steel, abrasive substances, and alloys that have enhanced hardness due to dispersion [100].

PM₁₀ and PM_{2.5} samples can be obtained in filters made of quartz [26,29,77,78], fiberglass [25,27,35,76], Teflon [16,78], polyurethane [28], in special bags [74], or in biofilters, such as leaves of some trees [51]. Among these, quartz and Teflon filters of the Whatman brand stand out as they were the most used in the studies analyzed. Teflon is one of the most common filters: durable, low-cost, and with a good collection efficiency for PM of all sizes. They are hydrophobic as well, making them useful for humid environments. On the contrary, these filters have limitations for analysis of trace metals and volatile organic compounds (VOCs), as they can interfere with the measurement. Quartz filter are hydrophobic, provide good collection efficiency for PM of all sizes, and are transparent, making them useful for the analysis of trace metals and VOCs, as well as for optical particle counting. They are more expensive than Teflon filters, but also more durable and able to be used in multiple tests.

Among the air volume capturing instruments, high volume capturers [29,77], low volume capturers [26], impactor type collectors [79,80], and Anderson and Envirotech brands stand out. Many authors use the sampling methodologies established by the EPA [5]. Ion chromatography (IC) and atomic absorption spectroscopy (AAS) are two techniques that can be used to separate and quantify ionic species and metal ions, respectively. IC can separate and quantify a wide range of ionic species (e.g., anions, cations, acids, and bases) in a sample and can provide high sensitivity and accuracy for trace level analysis. AAS can provide high sensitivity and accuracy for trace level analysis of metal ions and can detect and quantify a wide range of metals in a sample. Both IC and AAS can be used for both qualitative and quantitative

Inductively coupled plasma mass spectrometry (ICP-MS) is a highly sensitive and accurate technique for trace element analysis. It can detect a wide range of elements simultaneously and is particularly well suited for multi-element analysis and quantification of elements in the low parts-per-billion range. ICP-MS works by ionizing the elements in a sample and then measuring the mass of the resulting ions using a mass spectrometer.

The main detection method among the selected articles was inductively coupled plasma mass spectrometry (ICP-MS). Atomic absorption spectroscopy (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) are also highlighted [101] asserts that INAA technologies (instrumental neutron activation analysis) and IPAA (instrumental proton activation analysis) present more accurate results regarding the concentration of metals in PM samples. There are other useful technologies to characterize metals in aqueous samples which are not included among the methods selected by the scientists' authors of the selected articles, such as: anodic stripping analysis, cold vapor absorption spectrometry, among other methods developed in the Standard Methods.

Geographic Information Systems (GIS) can be a powerful tool for particulate matter (PM) characterization, as they allow for the integration of spatial data with air quality information. With GIS, PM data can be mapped and analyzed to identify patterns and relationships between air quality and various factors, such as land use, demographics, and sources of emissions. One of the main advantages of using GIS for PM characterization is the ability to visualize data in a spatial context. This allows for the identification of hot spots of PM pollution, which can be related to specific sources of emissions or environmental conditions. For example, areas with high levels of PM pollution may be associated with heavy traffic or industrial activities. GIS can also be used to model the dispersion of PM in the atmosphere, taking into account meteorological conditions and other factors that can affect air quality. This information can be used to identify areas that are most vulnerable to PM exposure, as well as to predict the potential impacts of changes in land use or emissions sources [102]. In addition, GIS can be used to integrate PM data with other environmental and health information, such as demographic data and hospitalization records. This allows for the analysis of the relationships between PM exposure and health outcomes, which is essential for understanding the public health impacts of air pollution.

5. Conclusions

The Proknow-C method establishes a suitable route for a comprehensive literature collection of a very significant issue with impact elsewhere, PM pollution, which concentrations vary according to site characteristics (temperature, location, nearby sources, cortical material, etc). This review is very important because atmospheric pollution is of public interest since it seriously affects the public health and the quality of the environment, with many diseases associated to PM. Ions occupy a large fraction of the total composition of PM samples, while metals occupy a small fraction, but their presence in the pollutant increases its toxicity and constitutes a danger to public health. Sulfate, nitrate, and ammonium ions are of great importance in the study of PM₁₀ and PM_{2.5} because their concentrations are relatively high, and they also affect visibility and cause multiple diseases. It was found that

the characterization techniques mostly used for metals are atomic absorption and plasma induction; while for ions is ion chromatography. In general, the atmospheric contamination phenomena are aggravated in the cold seasons elsewhere.

Among the selected articles, only ions that were soluble in water (WSIS) were found to be characterized, this is because they are the most relevant in the composition of PM, the IS occupy between 40 and 50% of the mass in the analyzed samples by scientists [70]. The most studied ions are sulfate (SO_4^{-2}), nitrate (NO_3^{-}) and ammonium (NH_4^+), likewise, the fraction of these ions occupy between 70–80% of the total concentration among the IS present in the PM [84].

Measuring the concentrations of ions and metals in PM samples is important because it provides valuable information about the quality of the air, avoiding public health issues. Knowing the concentrations of these components in PM samples, results can help to determine the sources and causes of air pollution, which is essential for developing effective control strategies to reduce exposure to harmful particles. Additionally, by monitoring the levels of ions and metals in PM, public health authorities can assess the potential health risks associated with exposure to these particles, and make informed decisions to protect public health.

PM is a major contributor to air pollution and can have serious impacts on human health. Measuring PM is important for understanding its sources, distribution, and impacts on the local population for other similar scenarios. This research also summarizes the steps involved in measuring PM. The first step in measuring PM is to select appropriate sites for the measurements. Once the sites have been selected, filters can be deployed. Sampling should be conducted following a standardized protocol, such as the one described by the World Health Organization or the U.S. Environmental Protection Agency. The protocol should specify the duration of the sampling, the flow rate of the air through the sampler, and the type of filter to be used. Data should also be collected on relevant environmental parameters, such as temperature, relative humidity, and wind speed and direction. After the sampling period, the filters should be removed from the samplers and sent to a laboratory for analysis. The filters can be analyzed using techniques such as ionic chromatography or atomic absorption spectroscopy, depending on the specific goals of the measurement. These techniques will provide information on the composition of the PM, including the presence of inorganic ions and metals. The data collected from the PM measurements and laboratory analysis should be analyzed to determine the concentration of PM, as well as its sources and composition. This information can be compared to air quality standards and used to identify areas with elevated levels of PM pollution. The PM data can also be mapped using Geographic Information Systems (GIS) to visualize the distribution of PM in the valley and to identify hot spots of PM pollution. By measuring PM and following a standardized protocol and using the appropriate techniques, it is possible to obtain a comprehensive picture of PM in a city in order to develop targeted strategies for reducing its impacts.

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