FOCUSED REVIEW



Recent Developments in the Determination of PM_{2.5} Chemical Composition

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Received: 21 January 2021 / Accepted: 16 March 2022 / Published online: 6 April 2022 © The Author(s), under exclusive licence to Springer Science+Business Media, LLC, part of Springer Nature 2022

Abstract

Fine particulate matter (named $PM_{2.5}$) has become a prominent and dangerous form of air pollution. The chemical composition of $PM_{2.5}$ mainly includes inorganic elements, water soluble ions, elemental carbon (EC), organic carbon (OC), and organic compounds. The detection method for inorganic elements mainly includes X ray fluorescence, inductively coupled plasma-atomic emission spectrometry, and inductively coupled plasma mass spectrometry. As for water soluble ions, ion chromatography is the most common detection method. EC and OC are usually detected by carbon analyzer. The organic compounds are determined by gas chromatography–mass spectrometry and liquid chromatography–mass spectrometry. In this paper, the merits and drawbacks of each analytical methods for the determination of $PM_{2.5}$ chemical composition are summarized. This review also includes our discussion on the improvement of the analytical accuracy for the determination of $PM_{2.5}$ chemical composition owing to the development of reference materials.

Keywords Air pollution \cdot PM_{2.5} \cdot Chemical composition \cdot Source appointment \cdot Reference material \cdot Analytical accuracy

Air pollution is a global problem and thus has been attacted more attention since 1930s. In 1952, the Big Smoke in London makes people concentrate on particulate matters (PM), which is divided into two kinds according to aerodynamics diameter (D_p). The particles with $D_p \ge 2.5 \ \mu m$ are called coarse particles, while others with $D_p < 2.5 \ \mu m$ are defined as fine particle matters and also named as PM_{2.5}.

The primary sources of $PM_{2.5}$ are traffic, industry, domestic fuel burning, and natural sources from volcanos, soil dust, and sea spray particles etc. Generally, particles which come from the burning of fossil fuel are considered as the major source of $PM_{2.5}$. The particles exhausted by nature and human activities are defined as primary particles (Streets et al. 2003). Another source of $PM_{2.5}$ is the growth of condensable vapors in the atmosphere, which is called new particle formation (NPF)

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(Kumala 2003). There are four different modes of NPF according to the particle diameter: nucleation mode, aitken mode, accumulation mode, and coarse mode. Particles with nucleation mode come from the condensation and coagulation of low volatility compounds (Kumala 2003), and then grow to aitken mode, accumulation mode, and coarse mode. The mechanism of particle growth is generally acknowledged in three ways: H₂SO₄-H₂O nucleation, H₂SO₄-H₂O-NH₃ nucleation and ion nucleation (Kirkby et al. 2011). The diameter of particles increases 40~100 nm in a few hours under high concentration of gaseous pollutant and strong oxidative (Wiedensohler et al. 2012). With the size growth of the particles, several chemical reactions such as photocatalytic reactions (Griffith et al. 2013) and free radical oxidation reactions (Schaefer et al. 2015) may occur, which makes the chemical composition of PM_{2.5} more complex. To determine the chemical composition of PM_{2.5} is significant to understand the mechanism of NPF. The atmosphere and regional climate may be influenced by $PM_{2.5}$. Atmospheric visibility is reduced by $PM_{2.5}$ since it increases scattering and absorption coefficients of air. With the concentration of PM_{2.5} increasing, the cyclone in the East U.S. is continually extended during the last 40 years (Tai et al. 2010). Besides the influence on the atmosphere and climate, PM_{2.5} also seriously effects on the human health, especially respiratory system and cardiovascular system, even at PM_{2.5}

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levels below the current standards (deSouza 2021). Statistical data showed that the number of SARS-CoV-2 infections cases and deaths increased significantly along with increasing levels of PM_{2.5} in five regions of the USA, Los Angeles, New Mexico, New York, Ohio, and Florida (Meo et al. 2021). Heavy metals (e.g. Pb and Hg) in PM25 may cause asthma and heart failure (Prendes et al. 1999). Polycyclic aromatic hydrocarbons (PAHs) (Shen et al. 2019) and heavy metals in PM_{25} are considered as the main reason for the increase risk of cancer. Determination of PM2 5 chemical composition is useful to evaluate the risk of PM₂₅ on human health. The PM₂₅-related health impact and corresponding economic losse for both society and individual were estimated, which can provide insightful observations for policy makers to keep the social equality, welfare and environment conservation. In the European Union, Directive 2008/50/EC has been enforced to monitor and reduce the contamination levels of PM2 5. Based on the information of PM25 chemical composition, source apportionment for pollution can be clearly identified, which is helpful to effectively control the air pollution (in't Veld et al. 2021).

In light of the reasons mentioned above, it is essential for the determination of $PM_{2.5}$ chemical composition. In this paper, the analytical methods for the determination of inorganic elements, water soluble ions, element carbon and organic carbon, organic compounds in $PM_{2.5}$ are carefully reviewed.

Sample Collection

 $PM_{2.5}$ needs to be collected in the atmosphere prior to analysis. Quartz fiber filter and teflon filter are commonly used in collecting $PM_{2.5}$. Quartz fiber filter has outstanding performance of high temperature resistance and therefore is suitable for the analysis of element carbon, organic carbon, organic compounds, and water soluble ions. In order to reduce the background of organic compounds, quartz fiber filter needs to be baked for a few hours under temperature higher than 300°C before used. Considering some inorganic elements (e.g. Al, Si and Fe) are inherently reserved in quartz fiber filter, it is not suitable to use quartz fiber filter for the analysis of inorganic element. Teflon filter is usually used in determination of inorganic inorganic elements and water soluble ions. The treatment of teflon filter is similar to quartz fiber filter.

Detection Methods for PM_{2.5} Chemical Composition

Determination of Inorganic Elements in PM_{2.5}

Several inorganic elements are extremely harmful to human health. X ray fluorescence (XRF) is a technique

used to characterize inorganic elements by measuring the fluorescence emitting from a chemical compound subjected to continuous X-ray radiation. XRF has the advantages of relatively easy operation and low cost of sample preparation, and thus is widely used for the determination of major and trace inorganic elements in several kinds of material, e.g. rock, mineral, and sediment. For the analysis of PM_{2.5}, XRF is usually used to detect heavy metals (e.g. Pb, Cu, and Cd) with detection limits between 1 and 100 $ng cm^{-2}$ (Gemenetzis et al. 2006). For example, 18 kinds of inorganic elements (e.g. Zn, As, and Sr) determined using energy dispersive X-ray fluorescence (ED-XRF), which results have an uncertainty between 5% and 15%, was adoped for source appointment of air pollution (Owoade et al. 2016). The ED-XRF system used had detection limits (mg/g) as shown in parenthesis: Na(2000), Mg(1000), Al(600), Si(300), P(120), S(30), Cl(10), K(150), Ca(80), Ti(25), Mn(0.5), Fe(15), Zn(0.8), As(0.6), Br(0.6), Rb(0.5), Sr(0.5) and Pb(1.7). Synchrotron radiation X-ray fluorescence (SR-XRF) is another XRF with synchrotron radiation used as excitation source. With main advantage of high sensitivity (detection limits in the range of $ng m^{-3}$), SR-XRF has proved to be a powerful tool to quantify the major, minor, trace constituents, which results were correlated with possible sources (López et al. 2011). XRF has the advantages of high speed, high precision, and simple sample treatment.

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) is another technique used for detecting inorganic elements. Using ICP-AES, it proved that heavy metals in PM_{2.5} are closely related with industrial production. For example, the concentrations of Cr, Co, Ni, As, and Sb in Shandong province were the highest in China because of the large amount of industries in this province (Liu et al. 2018). Alternatively, inductively coupled plasma-mass spectrometry (ICP-MS) is an analytical method for determination of inorganic elements in PM2 5. For example, Cu, Pb, Se, and As were found to be the highest inorganic elements in PM2 5 sample collected in Mountain Tai by using ICP-MS, and these inorganic elements mainly come from coal-fired power plants (Qie et al. 2018). With ICP-MS, 23 kinds of trace inorganic elements in PM_{2.5} sample collected in south Taiwan were detected, and their concentrations in cold season were higher than those in hot season (Hwang et al. 2018). The method detection limits were in the range 1.83×10^{-2} –24.3 µg L⁻¹ for all selected trace elements. Briefly, both ICP-AES and ICP-MS are highly effective analytical techniques. ICP-AES allows for a wide range of elements with major concentrations down to part per billion. With MS as detector, ICP-MS provides a lower detection limit of part per trillion.

Determination of Water Soluble lons in PM_{2.5}

Water soluble ions are major composition of PM2 5. Water soluble ions have strong hygroscopicity, which may influence the formation of cloud and thus the climate (Hassanvand et al. 2014). Atmospheric visibility is reduced by water soluble ions in PM_{2 5}via scattering sunlight (Christine et al. 1991). In addition, water soluble ions may form secondary aerosol inorganic ions (SAII) (Sharma et al. 2007). Resultly study showed heavy metals play an important role in the genotoxicity of PM_{2.5} (Kogianni et al. 2021). Ion chromatography has the advantages of high speed and good separation efficiency, it thus is widely used for the determination of water soluble ions in PM2 5. For example, a study on water soluble ions in PM2 5 using ion chromatography, with detection limit of each ion lower than 0.015 μ g m⁻³, showed that the secondary sulfate was ascribed to aqueous phase sulfur oxidation promoted by high concentration NO₂ and high relative humidity (Guo et al. 2019). Seasonal variability of NH_4^+ and NO_3^- is governed by dissociation of NH_4NO_3 under high temperature in summer (Qiao et al. 2019).

Besides ion chromatography, atomic absorption spectrophotometry (AAS) is another analytical method for determination of water soluble ions. The principle of AAS is absorption of specific frequencies of light by atoms, and thus AAS can only analyze metal ions. Nessler spectrophotometry, selectfluor spectrophotometry, ferrithiocyanate spectrophotometry, and barium sulfate precipitation are traditional analytical methods for NH₄⁺, F⁻, Cl⁻, and SO₄²⁻. These methods need complicated sample preparation and thus are not very popular in the detection of water soluble ions in PM_{25} . Several metal ions in PM_{25} can be used as a marker for source apportionment of air pollution. For example, Ca²⁺ and Mg²⁺ can be used as a marker of construction dust (Owoade et al. 2016), since the use of cement is the only source of Ca²⁺ and Mg²⁺ in an inland city. F⁻ can be used as a marker of coal burning (Galvao et al. 2019) because the only source of F⁻ is coal burning in a city. BaTiO₃ analyzed by synchrotron XRD was proved to be a vehicular marker formed during brake action. Maghemite (gamma-Fe₂O₃), pyracmonite $[(NH_4)_3Fe(SO_4)_3]$, ammonium perchiorate (NH_3OHClO_4) and potassium ferrate $(K_2Fe_2O_4)$ found in PM_{2.5} were proved to be markers of industrial activities (Galvão et al. 2020).

Determination of Elemental Carbon (EC) and Organic Carbon (OC) in PM_{2.5}

20–70% weight of $PM_{2.5}$ is composed of EC and OC. EC absorbs sunlight and thus heats the air, which directly contributes to global warming. EC has been considered as secondary factor contributing to global warming, followed with CO₂ (Jacobson 2001). OC mainly comes from exhaust of

vehicles and industry. The direct exhaust is called primary organic carbon, which can react with sunlight and oxidants (e.g. ozone and free radical) forming secondary organic carbon (Kelly and Fussell 2012). Besides, OC frequently derives from biomass burning (Zhang et al. 2010).

Carbon analyzer (e.g. DRI carbon analyzer, Sunset carbon analyzer) coupling with IMPROVE, NIOSH or EUSAAR2 thermal-optical protocols is widely used for the detection of EC and OC. Using either IMPROVE, NIOSH or EUSAAR2 protocol, the results of the total carbon (TC) were always the same, however the ratio of OC/EC was sometimes different because of different laser correction methods used (Bautista et al. 2015). How to choose a protocol depends on the actual situation, IMPROVE is a popular protocol, while EUSAAR2 protocol is mainly used in Europe (Vodicka et al. 2015). A typical limit detection for carbon analyzer is 0.33 μ g Carbon m⁻³ (Zhang et al. 2010).

Single particle aerosol mass spectrometry (SPA-MS) is a new technique for detecting the EC and OC in $PM_{2.5}$. Compared with traditional carbon analyzer, SPA-MS can determine the chemical composition of $PM_{2.5}$ in real-time without sample pretreatment.

Determination of Organic Compounds in PM_{2.5}

In recent years, more researches have been focused on the study of organic composition in $PM_{2.5}$ because of the high toxicity of organic compounds. It is difficult to accurately analyze all organic compounds in $PM_{2.5}$ because of the instability and low concentration of some complex organic compounds. PAHs are strong carcinogens and thus the risk of cancer arising from PAHs in $PM_{2.5}$ has been carefully studied (Shen et al. 2019). Dioxin is a typical PAH in $PM_{2.5}$, its high risk of carcinogenicity and mutagenicity has been intensively studied (Tsai et al. 2018). Besides PAHs, other organic compounds such as polychlorinated biphenyls (PCBs), fatty acid, and phthalate may participate in photochemical reaction forming more toxic compounds (Canonica et al. 2005).

Gas chromatography–mass spectrometry (GC–MS) and liquid chromatography–mass spectrometry (LC–MS) are two common methods for detecting organic compounds in $PM_{2.5}$. One significant advantage of GC–MS and LC–MS is good separation efficiency especially when a series of organic compounds with similar structures detected. GC–MS is used to detect small thermo-stable molecules with low boiling point. For the determination of PAH in $PM_{2.5}$, the recoveries were from 75% to 120% and relative deviation of six times duplicate was less than 10% (Shen et al. 2019). A GC–MS method was developed for simultaneous detection of 16 kinds of United States Environmental Protection Agency (US-EPA)-PAHs in $PM_{2.5}$, and the recovery obtained from urban dust SRM 649 A (NIST reference material) was between 63% and 102%. The results showed the concentrations of PAHs were increased with the use of wet wood for residential combustion (Guerrero et al. 2019). Another GC-MS method was developed for detecting the concentrations of PM2 5-associated non-polar organic compounds (NPOCs), including PAHs, n-alkanes, iso-alkanes, hopanes, and steranes. With this method, the size-specific distributions of NPOCs from 0.01 to 18 µm were also determined (Han et al. 2018). LC-MS has been applied to detect larger molecules (e.g. organosulfates) and thermo-unstable small organic molecules (e.g. amino acids) (Samy et al. 2011) in PM_{2.5}. For example, an LC-MS/MS method was developed for the detection of methylene blue active substances (MBAS) and linear alkylbenzene sulphonates (LAS) in PM25 sampled in a costal city, the limit of quantitications for LAS and MBAS were 2.2 ng m^{-3} and 45 $ng m^{-3}$, respectively. The results showed that the source of MBAS was anthropogenic origins and the source of LAS was sea surface (Becagli et al. 2011). Several methods based on GC-MS and LC-MS have been developed for detecting organic compounds in PM2 5, there are still some unknown organic compounds to be intensively studied. Also, complex sample preparation is needed before GC-MS and LC-MS analysis.

Alternatively, aerosol mass spectrometer (AMS) is a powerful technique for the direct study on atmospheric particles (Nash et al. 2006) and thus is widely used in the world. Using AMS, the organic component in the $PM_{2.5}$ can be efficiently ionized by electron impact, or chemical ionization. Subsequently, the ions are determined by a mass analyzer, such as time of flight, quadrupole mass filter, and quadrupole ion trap. Because complex sample preparation is not needed, AMS has potential application in the field of online determination of chemical composition in $PM_{2.5}$ and thus the source appointment of air pollution can be rapidly obtained.

Conclusions

 $PM_{2.5}$ is a common air pollutant seriesly effecting on human health. The chemical composition of $PM_{2.5}$ is complex mainly including inorganic elements, water soluble ions, EC, OC, and organic compounds. Accurate determination of chemical composition of $PM_{2.5}$ is useful for source appointment of air pollution, which is conducive to pollution control and environmental protection. Many methods are available for the detection of inorganic elements, water soluble ions, EC and OC in $PM_{2.5}$. However, comprehensive determination of organic compounds in $PM_{2.5}$ is still challenging although several methods based on GC–MS and LC–MS have been established. Therefore, several methods based on high resolution mass spectrometry should be developed for comprehensive characterization of organic compounds in PM_{25} . In the future, the accurate analytical method for determining PM2.5 chemical composition needs to be further developed. A reference material of PM2 5-like atmospheric dust material for accurate monitoring of anions and cations has been prepared (Charoud-Got et al. 2017) approaching the EN12341 definition of PM25 containing the ions mentioned in Directive 2008/50/EC. This reference material is very useful as a quality control sample for ensuring compatibility of results among PM25 monitoring studies and can be used to assess the accuracy of analytical methods used. Continuous efforts and achievements in the development of reference materials for monitoring other chemicals in PM_{2.5} would significantly improve the analytical accuracy for the determination of PM_{2.5} chemical compositions. Development of portable monitor achieving tempo-spatial patterns of chemicals in PM2.5 is also highly recommended to manage air pollution.

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