Single-Particle Analysis of Atmospheric Aerosols

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Atmospheric aerosols, produced as a consequence of different anthropogenic and natural processes, impart significant control over the global energy budget, climate, and human-environmental health. Their size varies across the nano-micrometer scale. Based on their origin, they may be classified into primary or secondary aerosols. Biomass burning, incomplete combustion of fossil fuels, volcanic eruptions, and traffic-related and wind-driven suspensions contribute to primary aerosol emissions. In contrast, gas-to-particle conversion within the atmosphere leads to secondary particle production. The study of atmospheric aerosols is vital to the field of atmospheric research. The dynamic nature (highly variable concentration composition and size with space and time) of aerosols makes them difficult to investigate. Today, aerosol research involves the application of various spectrometric and spectroscopic techniques. The single-particle analysis of aerosol is yet a challenge. The merits and demerits of various offline and online techniques used for aerosol research are discussed in a nutshell. Mass spectrometric techniques fail in distinguishing certain species.

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1. Introduction

Atmospheric aerosols are suspensions of liquid/solid particles in the atmosphere. Their size (diameter) varies from a few nanometers to tens of micrometers and they play a significant role in controlling the hydrological cycle and energy balance of the Earth. They can manipulate the global climate and local air quality [1]. Both anthropogenic and natural activities contribute to aerosol emissions. Aerosols are broadly classified into primary and secondary aerosols. Primary aerosols are directly emitted into the atmosphere from the source. In contrast, secondary aerosols are formed by the oxidation of gaseous precursors that nucleate to form particles or condense onto an existing particle [1]. The residence time of aerosols in the atmosphere varies from a few seconds to a month. The lifetime of aerosols in the atmosphere is determined by their particle size, chemical composition, and local meteorology [2]. Meanwhile, the bulk particle composition is determined by their source and different aging processes in the atmosphere [2][3]. The atmospheric transport can alter their size, phase, structure, and chemical composition. The factors that govern this modification involve coagulation, gas–particle partitioning of semi-volatile species, heterogeneous reactions of particles with trace gases, and aqueous-phase processing [1]. The dynamic nature of the physical and chemical properties of the aerosols makes their study challenging.

A single aerosol may host a staggering number of molecules. The number of aerosols per unit volume (concentration number) and the total mass of the aerosols per unit volume (mass concentrations) vary spatially and temporally. It is reported that the particle concentration may range from 15 particles/cm^3 at remote locations to 10^5 particles/cm^3 or greater in urban areas. The mass concentrations of PM$_{2.5}$ particles range from 1–50 mg/m^3 in rural areas to ~200 mg/m^3 in heavily polluted urban areas [4]. PM$_{2.5}$ particles are fine, inhalable particles having an aerodynamic diameter of ≤2.5 µm. They can scatter visible light efficiently; thus, high PM$_{2.5}$ levels in the atmosphere often lead to reduced visibility/haze events. The available literature on atmospheric aerosols mainly focuses on characterizing their bulk properties. This can be attributed to the various challenges faced during characterization at the single-particle scale.

A wide range of instruments are available for both the offline and online analysis of aerosols. Offline techniques involve the collection and transportation of samples from the field to the laboratory. In the case of online analysis, the samples are measured in real time. Hence, offline analysis is often time-consuming when compared with online analysis. However, a single practical technique is still not available for obtaining all the required information. For example, if researchers are interested in molecular and structural analysis, offline techniques are superior to online techniques. Likewise, online techniques are better suited for monitoring the modification in chemical composition on short time scales [5]. Some offline and online techniques for carrying out the single-particle analysis of aerosols are listed in **Table 1** [6].

Table 1. Some offline and online techniques used for the single-particle-level characterization of aerosols.

Offline Techniques	Online Techniques
Scanning electron microscopy–energy-dispersive X-ray (SEM-EDX) Micro-Raman spectroscopy (MRS) High-resolution transmission electron microscopy (HRTEM) X-ray photoelectron spectroscopy (XPS) Nano-scale secondary ion mass spectrometry (Nano SIMS) Time-of-flight SIMS (TOF-SIMS) X-ray absorption fine structure spectroscopy (XAFS) X-ray absorption near-edge structure (XANES) spectroscopy Electron energy loss spectrometry (EELS) Proton-induced X-ray emission (PIXE) Single-particle inductively coupled mass spectrometry (SP-ICPMS) Laser microprobe mass spectrometry (LMMS)	Aerosol time-of-flight mass spectrometer (ATOFMS) Laser-induced breakdown spectroscopy (LIBS) Aerosol mass spectrometry (AMS)

The chemical composition of an aerosol is highly sensitive to the surrounding environment. Thus, during sample processing, preparation, or analysis, there is a possibility of the contamination/alteration of the chemical species in the aerosols. SEM-EDX and HRTEM are widely used for the analysis of fine atmospheric particulates, but the application of a vacuum at the analysis stage results in the loss of volatile compounds [7][8]. The spectrometric techniques, such as Nano SIMS, TOF-SIMS SP-ICPMS, LMMS, ATOFMS, and AMS, provide high-resolution information on the distribution of chemical species over the aerosols. However, these techniques are destructive in nature. There are non-destructive spectroscopic techniques, such as XPS, PIXE, and EELS, which provide the scope for monitoring ultrafine particles [6][9][10]. However, certain limitations exist with these techniques; for example, in the case of XPS and EELS, it is extremely difficult to interpret the spectra generated from the analysis, while the PIXE technique falls short when it comes to the quantification of low-Z elements [6][11]. In the case of XANES and XAFS, even though they offer high detection sensitivity, their limitation lies in the requirement of a sophisticated synchrotron X-ray source [12]. LIBS is an effective tool for monitoring aerosols at single-particle scale. It does not require sample preparation and allows real-time measurement of the particle mass and composition. However, a drawback associated with this technique is its susceptibility to large interference effects [6]. In this context, MRS appears to be a potential candidate over other techniques for the characterization of atmospheric aerosols at single-particle scale. It does not require tedious sample processing steps, and also provides rapid information on the chemical composition, phase, and crystal orientation of particles. MRS can also determine the different oxidation states of chemical species distributed over the aerosols. However, the limitation of the MRS technique results from its inability to monitor particulates in the nanoscale range [6][13].

The mixing states of aerosols are classified into external or internal mixtures. The aerosols in internal mixtures contain various chemical species with the same mixture composition throughout a single particle. External mixtures are composed of aerosols that only host a single pure species within a particle [14]. These mixing states can be effectively monitored using spectroscopy. Further, due to their non-destructive nature, the samples could be used for other analyses involving different instrumentation techniques [15]. Molecular specificity, the ability to neutralize the spectral interference from water, and accessibility to the visible spectrum’s low-frequency region make Raman spectroscopy an ideal tool for aerosol characterization [16].

Techniques such as attenuated total reflectance Fourier transform infrared spectroscopy (ATR FT-IR), diffuse reflectance FT-IR, or optical microscopy coupled with FT-IR (micro-FT-IR) can also be used for the analysis of the chemical composition of aerosols. However, their application is not recommended in the presence of water due to the strong absorbance interference. Thus, they are limited to bulk particle analysis [17]. In this context, micro-Raman spectroscopy (MRS) offers immense scope for analysis at a single-particle scale. The advantages of MRS include a non-intrusive investigation at ambient temperature and pressure, and minimal sample preparation convenience. Lately, the MRS technique has become a highly preferred one for studies concerning the composition and mixing state of individual aerosols [18]. Raman imaging provides spatial distribution data about the functional groups in an aerosol, rather than only producing vibrational frequencies of functional groups. MRS is also very effective in measuring the acidity of aerosols at a single-particle scale [17][19]. Of late, surface-enhanced Raman spectroscopy (SERS) has gained high interest in the single-particle analysis of atmospheric aerosols. This technique provides strong enhancements in the Raman signal owing to the localized surface plasmon resonances (LSPRs) activated over small (nano-scale) spatial regions of the substrate [20]. Herein, researchers present a detailed overview of Raman spectroscopy’s application as a tool for the characterization of atmospheric aerosols.

2. Ambient Aerosol Studies

A large number of studies are available based on the characteristics of ambient aerosols. However, most of them are limited to bulk analysis. To obtain insights into the effect of aerosols on climate, human, and environmental health, it is recommended to carry out a single-particle analysis of ambient aerosols [6]. Due to the dynamic nature of ambient particulates, it is always a challenge to decipher their physical and chemical characteristics. Micro-Raman spectroscopy (MRS) provides the opportunity to rapidly analyze aerosol particles at the microscopic scale. The non-destructive nature of this technique [21] makes it a potential tool for studying ambient aerosols.

Aerosols may contain noxious components that are detrimental to human health [22]. The capability of an airborne particle to cause health issues is directly correlated with its size. Particles that have a size of <10 µm are found to cause the most health problems. This is due to their ability to penetrate deeply into the lungs and even into the bloodstream. Thus, size-segregated particle analysis is necessary to identify the role of particulates in causing respiratory and cardiovascular dysfunction.

Earlier studies have established a potential link between soot exposure and respiratory and cardiovascular diseases. MRS is a powerful technique for the size-segregated characterization of carbonaceous particulates. A study employed this technique to effectively characterize the different aerosol fractions obtained from an urban atmosphere [23]. The prevalence of soot particles (1323 cm^{-1} and 1582 cm^{-1}) in finer aerosol fractions, such as PM < 1 and 1–2.5 µm, was noted.

Oceans contribute significantly to the total aerosol concentration in the atmosphere [24]. However, limited information exists on the chemical composition of the emitted aerosols. The MRS analysis of the aerosols collected during a long (11,000 km) cruise between Chile and the USA indicated the dominance of long-chained organic compounds [25]. These compounds were identified by their intense doublet peaks occurring at ~2880 cm^{-1} and ~2850 cm^{-1} from C–H stretching.

Source apportionment studies focused on the long-distance transport of aerosols have effectively used MRS. A study over Kozani, Northern Greece [26] showed the existence of various mineral phases in the collected aerosols, such as calcite (1084, 710, 274, and 149 cm^{-1}), gypsum (1134, 1006, and 413 cm^{-1}), titanium oxide (627, 390, and 148 cm^{-1}), feldspar (510, 474,285, 265, 150, and 109 cm^{-1}), lepidolite (1573, 1347, 728, 382, and 288 cm^{-1}), and smectite (1379, 731, 385, 288, and 141 cm^{-1}). The presence of lepidolite (mica) and smectite (clay) was associated with the long-distance transport of Saharan dust.

Acid precipitation is mainly influenced by the availability of acidic components (mainly sulfate and nitrates) in fine atmospheric aerosols. It can occur over less polluted areas due to the long-distance transport of aerosols from industrialized regions. In this respect, MRS was effective in characterizing PM_1 samples over a mountain environment subjected to acid precipitation because of the transport of aerosols from industrialized regions [27]. The Raman spectrum could successfully reveal the dominance of sulfate ion (~1000 cm^{-1}) species in the aerosols. Another study used this technique to monitor aerosol samples collected from an urban location [28]. The Raman spectrum indicated the dominance of particles with metal oxides (Fe_2O_3) coated with carbonaceous species. The study further showed that altering the laser beam intensity can unveil the species distribution in a particle. For example, at a low beam intensity (5%), only G (1361.5 cm^{-1}) and D (1576.5 cm^{-1}) bands of carbonaceous species were noted, whereas at a high beam intensity (50%), the strength of bands (G and D) of carbonaceous species decreased, with a corresponding enhancement in the peaks of Fe_2O_3.

A large volume of studies worldwide have found that various ecosystems face a significant threat from microplastic pollution [29]. Most of these studies are mainly concentrated on marine and freshwater environments. Only a few studies are available indicating the contamination of air with microplastics [30]. The available literature has highlighted the use of the MRS technique to detect microplastics [31][32][33]. Recently, MRS was applied for the analysis of microplastics in air samples collected from a metropolitan area in Hamburg, Germany [34]. The spectra of collected microplastic fragments and fibers revealed polyethylene terephthalate (PET) and polyethylene (PE). Different microplastics can be identified from a pure sample or a mixture from their characteristic peak. The peaks at 1000 cm^{-1}, 1720 cm^{-1}, 1059 cm^{-1}, 695 cm^{-1}, and 402 cm^{-1} correspond to PS, PET, PE, polyvinyl chloride (PVC), and polypropylene (PP), respectively [35].

3. Surface-Enhanced Raman Spectroscopy (SERS)

Traditionally, the most preferred methods for the chemical characterization of aerosols involve the use of mass spectrometric techniques. This is due to their capability in detecting species present at trace (ng/L) concentrations [36]. These techniques are, however, costly and time-consuming. Because of the dynamic nature of the aerosols, it is always better to analyze samples rapidly after collection. The mass spectrometric technique often falls short for the rapid analysis of samples, as it requires pre-sample processing [36]. These steps may alter the properties of the aerosols. This highlights the necessity of a sophisticated technique that is fast and does not require pre-sample processing. With surface-enhanced Raman spectroscopy (SERS), it is now possible to detect species at the atto-gram to femtogram level in particles [18]. In the SERS technique, the signals emanating from the analytes are amplified to a great extent by the localized surface plasmon resonance (LSPR) produced by the nanoparticle framework in the substrate used for the analysis (**Figure 1**) [36].

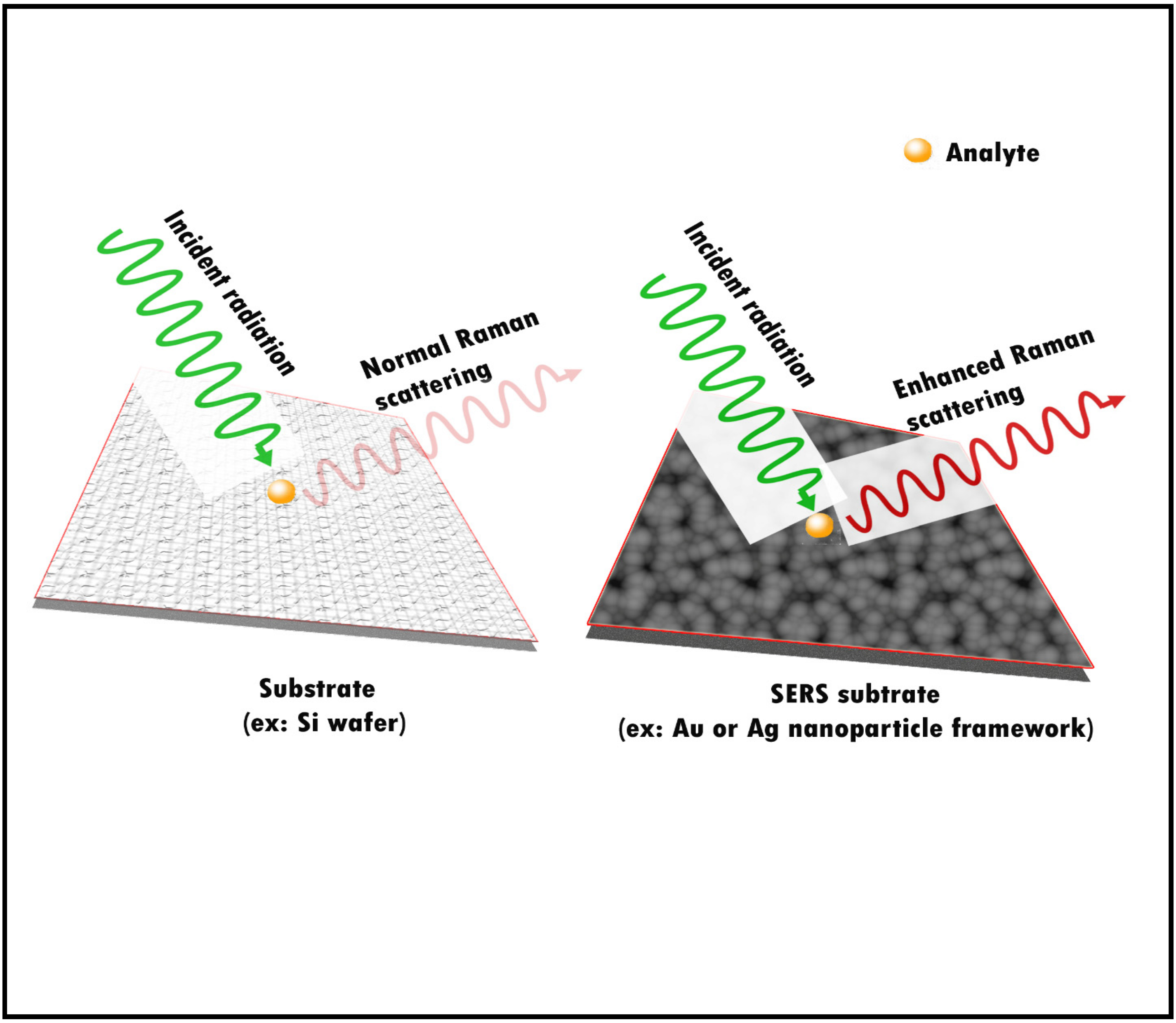

Figure 1. Schematic representation of surface-enhanced Raman spectroscopy (SERS).

Silver (Ag) or gold (Au) nanoparticle-coated quartz filters (SERS substrates) are often used for aerosol studies. A Au-coated quartz filter could detect the intraparticle distribution of functional groups ν_w (CH_2)–1022 cm^{-1}, δ (C–C)–1480 cm^{-1}, and ν_{as} (NO_3^-)–1370 cm^{-1}) over an SOA particle with 0.5 µm spatial resolution [18].

The amplification of the scattered signals is primarily attributed to the distribution of active sites (localized regions with enhanced electromagnetic fields) over the SERS substrate. The distribution of these over Ag- or Au-coated quartz filters is reported to be non-uniform. Hence, it can sometimes result in the weak enhancement of scattered signals, besides low measurement reproducibility [37]. Thus, better enhancement is only given by substrates hosting a uniform distribution of active sites over their surface. In this context, the commercially available Klarite substrate is recommended [37].

Some filters used to collect ambient aerosols include quartz, Teflon, polycarbonate, polyester, nylon, silver (Ag) foil, and aluminum (Al) foil [6][38][39][40]. A study showed that Ag foil can act as a SERS substrate by producing intense Raman peaks for species such as soot (1350 cm^{-1} and 1589 cm^{-1}), nitrates (1045 cm^{-1}), and sulfates (978 cm^{-1}) in samples [41].

Haze events often result from high concentrations of sulfate–nitrate–ammonium (SNA) components in the fine aerosol fractions (<2.5 µm). However, understanding their mixing at a single-particle scale is a challenge. Therefore, a highly sophisticated and non-destructive technique is necessary to obtain insights into their distribution over an aerosol. The SERS technique was employed successfully to delineate the distribution of SNA species over aerosol particles [42]. The identified salts included $3(NH_4NO_3)\cdot(NH_4)_2SO_4$, $2(NH_4NO_3)\cdot(NH_4)_2SO_4$, and NH_4NO_3. Of late, Ag-SiO_2 nanocomposites have been found to be a highly sensitive SERS substrate for monitoring anthropogenic VOC mixtures (toluene, benzene, chloroform, and acetone) at trace levels under room temperature [43]. Although the SERS technique is slowly gaining interest in atmospheric research, this method seems to be largely underutilized when considering its huge potential.

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