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Methodology to measure atmospheric nanoparticles charge *

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ABSTRACT

Atmospheric pollution has become a key aspect for sustainable development world-wide. Lack of measurements of atmospheric nanoparticles properties at different geographic locations limits the understanding of the role atmospheric particulate matter plays in multiple biophysical and environmental processes and its corresponding risks for human beings. This study presents a method to measure atmospheric primary nanoparticle, secondary nanoparticle and microparticle data. Moreover, a process for samples characterization is proposed combining different spectroscopy techniques.

- The method allows researcher to collect, measure, store and characterize atmospheric nanoparticles properties including their electric charge.
- A specific sample characterization is proposed, based on different techniques such as TEM and RAMAN spectroscopy.
- The outcomes of the approach give science the chance to study new themes such as the importance of particulate matter charge in transmission of infectious respiratory diseases; the role of electric charge in pollutants deposition in the respiratory tract; the link between electric atmospheric charge of nanoparticles and meteorological variables.

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Abbreviations: ELPI©+, (Electric Low-Pressure Impactor); Di, (Stokes diameter); D50%, (Aerodynamic diameter); Fa, (femtonampere); fC, (fentocoulomb); mbar, (millibar); nm, (nanometre); pt/cm^3 , (particulates per cubic centimetre); $\mu m^2/cm^3$, (squared micrometers per cubic centimetre); ml, (millilitre); TEM-EDX, (Transmission Electronic Microscopy- Energy Dispersive X-Ray); RAMAN, (RAMAN spectroscopy).

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Method details

Introduction

Atmospheric particulate matter measurement is nowadays a key issue in relation to multiple scientific topics such as atmospheric physics, public health, climate change, biometeorology, ecology, clouds formation [1–5]. This methodology was developed under the framework of the COST Action 15,211 Electronet, in Working Group IV. One of the actions of this WG-IV consisted on measuring atmospheric nano and micro size particulate matter data in the city of Santander (Northern Spain). An electrical low-pressure impactor (ELPI®+) was facilitated by Dekati Limited Company, through the Spanish company SOLMA Solutions, to the Geobiomet Research Group at the University of Cantabria in the frame of the indicated COST scientific Action.

Charge of atmospheric particles can be a key parameter to understand the connections between atmospheric processes and environmental health. To date, information on potential interactions between global electric circuit and living organisms in symbiotic ecosystems is limited [6–9]. The need of common language amongst scientific disciplines to work on this topic under a multidisciplinary approach is needed, even more when considering that human being is a bioelectric organism. Some of the main outcomes of COST Action 15,211 indicated that specific respiratory, cardiovascular, infectious and neurodegenerative diseases [10–12] may be related, through complex pathways, to atmospheric electromagnetic fields. Even some mental disorders [13,14] were theoretically associated to the electromagnetic component of the environment.

Several dimensions associated to the measurement of atmospheric nano and microparticles are summarized in Fig. 1. Meteorological factors play a key role on spatial spreading of atmospheric nano and microprticles. In this sense, electrical properties of particles relate with other meteorological variables such as air humidity, temperature, and atmospheric pressure. Measurements of charge of particulate matter can facilitate to atmospheric scientists the classifications creation of weather types and air masses based on electrical properties at regional scales attending to different geographic landscapes [15,16] in other to associate electrical environment [17] to different regions .

According to Hsiao et al. [18], fine particles found in the air are likely a transmission media for influenza virus. Aerosols exhaled by infected population can use particulate matter existing in the air as a vector to transmit the disease [19,20]. In this sense, charge as a nanoparticle property, can play a significant role in transportations of microorganisms such as viruses and bacteria [21], which can harm human health.

Size, shape, morphology, and chemical composition of nanoparticles are important in the deposition of particulate matter in the respiratory tract of people. The study developed by Fdez-Arroyabe et al. [22] indicates that more particles with smaller aerodynamic

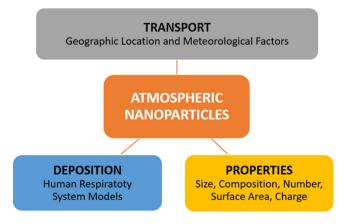


Fig. 1. Main aspects related to the measurement of atmospheric particles.

Table 1



Fig. 2. Electrical low-pressure impactor (ELPI®+) with 14 channels vertical sampling line.

Stage	D50% [μm]	Di [µm]	Number min [1/cm ³]	Number max [1/cm ³]	Mass min [µg/m ³]	Mass max [mg/m ³]
15	10					
14	5.3	7.3	0.10	1.7E+04	11	3400
13	3.6	4.4	0.10	3.0E+04	4	1300
12	2.5	3.0	0.16	5.2E+04	2.3	730
11	1.6	2.0	0.3	9.7E+04	1.3	400
10	0.94	1.2	0.6	2.0E+05	0.6	195
9	0.6	0.75	1.2	3.9E+05	0.3	85
8	0.38	0.48	2	6.8E+05	0.12	38
7	0.25	0.31	4	1.2E+06	0.06	17
6	0.15	0.19	6	2.0E+06	0.03	7.7
5	0.094	0.12	12	3.7E+06	0.01	3.2
4	0.054	0.071	21	7.0E+06	0.004	1.3
3	0.030	0.040	42	1.4E+07	0.0015	0.47
2	0.016	0.022	90	3.0E+07	0.0005	0.16
1	0.006	0.010	240	7.9E+07	0.0002	0.03

Nominal impactor specifications for each channel according to ELPI©+ manual.

diameter deposit at alveolar region according to the ICRP 94 model [23]. This research focused on Brownian motion and only samples with 6 nm to 380 nm range were characterized. This modelling was used to estimate deposition in the three main regions of the respiratory tract. Predicted total and regional deposition by the ICRP model [24] was estimated by channel as a function of particle size.

The method proposed in this article considers the hypothesis of nanoparticles charge being a potential risk factor for human health. Its measurement may help us to understand the deposition mechanism of harmful particles in different areas of the human respiratory system. The main objective of this article is to show the methodology followed to measure atmospheric nanoparticles properties, mainly electric charge, using an ELPI®+ device. This can help to study how atmospheric electric fields influence health of living organisms and show its potential use in different research fields such as atmospheric physics, climatology, environmental health, ecology, epidemiology or biometeorology.

Materials and method

Site description and settle down

The devices were installed on the roof of the Faculty of Philosophy (Fig. 2), separate from the perimeter wall of the roof. The device was installed at point N 43° 28′ 25.07 and W 3°47′ 56.54, not far from the shoreline of the Cantabric Sea. The building is located less than 2 km from the sea coast, and is exposed to inclement weather, winds, fog and precipitation. This site was selected after considering the absence of any chimney or heating or electrical sources around in order to prevent quality of records. The installations fed with fossil fuel (diesel and natural gas) in the building and in the adjacent ones remained off during the measurement period. Traffic in the area was lower than normal when the measurements were recorded, during the summer period. At this time Faculties activity is very reduced compare to the rest of the year.

Instrumentation

An electrical low-pressure impactor (ELPI $(\mathbb{R}+)$) is a real time and offline analysis instrument to measure particles from 6 nm to 10 μ m with 14 size fractions (Table 1). Measurements were recorded from 14 different channels from 4th to 30th of July 2018. Extraction

MEASUREMENT STRATEGY Based on 1 minute cycle					
30 seconds	30 seconds				
	Mode 2				
Mode 1	Mode 2				

Fig. 3. Measurement strategy based on 1-minute cycle and two modes.

of samples from the device was weekly. Real-time measurement gives number concentration, size distribution, surface area, mass, volume, electric current and electric charge. It takes data from particulate matter within its size range as a whole measurement, without distinguishing its source and neither its chemical composition. Measurement devices were:

- An airtight and ventilated security box to insert the ELPI®+ device inside to protect it from meteorological conditions.
- An Air Pump. Main specification of air pump pressure under the first stage should be 40 mbar. According to manual specifications of ELPI (m, μ) , pump requirements are a minimum of 16 m³/h at 40 mbar abs., but it is recommended to work at 25 m³/h at 40 mbar.
- A collector cone to facilitate the inlet of air through a pipe to the electrometers.
- An electrical low-pressure impactor (ELPI®+) with 14 channels (Fig. 2). Each channel measurement includes Stokes diameter range Di (nm) and aerodynamic diameter D50% (nm), (Table 1).
- A laptop connected to the device and a communication card to access to the device remotely.

Particles are charged into a known charge level by the integrated corona charger before being directed to the cascade impactor. Inertial classification of particles based on aerodynamic size takes place for the 14 size fractions separately. Each electrometer channel detects the electric particle charge with a measurement range from 0.5 fA to 500,000 fA.

Measurement strategy

Table 1 presents ELPI®+ nominal impactor specifications.. Column *Stages* orders each channel range threshold, from smaller particles in channel 1 to bigger ones in channel 14. Each channel range threshold of ELPI®+ includes Stokes diameter (Di) and aerodynamic diameter (D50%) measurements.

The Stokes diameter is more accurate at our nanoparticle size goal [25,26], and was used to take electric charge measurement. However, the human respiratory tract model formula works with the aerodynamic diameter, so we took the equivalent unit of each channel number within each channel range threshold. The D50% or cut-off diameter is the size of particles collected with 50% efficiency on each impactor stage.

Inertial particle size classification takes in the cascade impactor along its 14 channels based on aerodynamic size [25]. The ELPI©+ strategy measurement was programmed based on 1-minute cycle (Fig. 3) using two modes of 30 s of periodic repetition during the entire data collection period. A 6-hour zeroing of the electrometer stages was programmed to each channel with ELPI©+ considering a typical ambient aerosol, it was taken a particle density of 1 g/cm³ to calculate the particles mass.

Mode 1 completes measurement of physical properties excluding electrical charge, with the corona charger turned on. Particles are charged as they enter the corona charger with a known charge level, are classified by size in each channel, and measurement of number concentration, volume, mass, size distribution, and surface area take place. The data selected for its relationship with the standardized exposure limit units were number concentration (pt/cm^3), mass (g), surface area ($\mu m^2/cm^3$) and size distribution (nm).

Mode 2 records direct measurement of the natural electrical charge of the particles in each channel. It involves having the corona charger turned off. The particles enter with their raw charge and they are classified by size in each channel. Current (fA) and electric charge (fC) are measured. Measurement in mode 2 has to last a minimum of 20 s to assure there is no artefacts in the process of measurement. Consequently, measurement strategy was to define 30 s for each mode.

Data preprocessing and samples

ELPI®+ real-time measurement was recording by second and each stage (one data per channel per second) including current and electric charge. A proper pre-impactor, vertical sampling line was used and ACTRIS aerosol in-situ sampling protocols as well as WMO-GAW guideline [27,28] were took into consideration. Data preprocessing consisted on testing the consistency of time series and cleaning the raw data collected by the ELPI®+ excluding missing data associated to the need of stopping the device weekly to

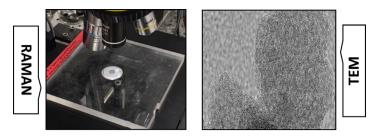


Fig. 4. A picture of the RAMAN device is on the left and a resulting image of TEM characterisation of multi-walled carbon nanotube at 5 nm scale on the right side (Geobiomet ©).

collect samples. The 10th and 90th percentiles were used to eliminate anomalous extreme values from the temporal series. Using the software R-Studio, statistical aggregation of raw data was carried out at four different temporal scales (10 min, 1 h, 6 h and 24 h).

The human respiratory tract deposition modelling was focused on the main regions of the respiratory tract: Head Airways (HA), Tracheobronchial (TB) and Alveolar (AL). The total and regional deposition percentages were calculated for the 14 channels with a particle size range from 6 nm to 10 μ m. It is important to emphasize that charge measured for each channel is associated only to the total human respiratory tract deposition as a whole.

Data collected indicated that negative charge was clearly associated with primary atmospheric nanoparticles in Channel 1 [6– 16 nm] and Channel 2 [16–30 nm] being mainly deposited in the alveolar region where Brownian motion of deposition is characteristic.

However, negative charge of nanoparticles of Channel 3 [30–54 nm] and Channel 4 [54–94 nm] was not son predominant. Lastly, exclusive positive charges were recorded for particles from Channel 5 [94–150 nm] to Channel 14 [5.300–10.000 nm]. Nanoparticles have shown an alveolar surface area deposition plateau with a size distribution range between 6 nm and 150 nm.

Samples collection and offline analysis

The collection substrates were aluminium foil, 25 mm in diameter, with a maximum thickness of 0.1 mm (ELPI®+ manual 1.55). This substrate is thin, smooth, and pore-free. The face of the impact collection substrate was greased to reduce the particle bounce effect. Real-time measurement allows users to know the amount of mass loaded on each channel before taking out the samples. The particles showed regular deposition on the surface substrate on visual observations. Collected samples were characterised with Transmission Electronic Microscopy (Jeol Jem 2100 with XEDS) and Raman Spectroscopy (T 64000, Horiba-Jobin-Yvon). Raman spectroscopy characterization kept the collected samples unchanged, allowing later characterizations with other techniques. TEM characterization involved removing the greased layer beneath and around the collected sample. Steps followed to prepare the samples for this technique were:

- 1. Cleaning of samples with 2 ml cyclohexane, with 2 ml ethanol and with 2 ml acetone.
- 2. Sample sonication for 10 min at 40 °C after each cleaning.
- 3. Drying samples for 24 h.
- 4. Storing dry power in unbreakable plastic containers with screw cap, inside zip clear plastic bag. Containers were labelled as nanomaterial and with hazard pictograms.
- 5. Before taking samples to the TEM characterization was compulsory to certificate (through RAMAN outcomes) that were free of any impurities.

Samples characterization

Raman Spectroscopy Service, and Transmission Electronic Microscopy Service coupled with EDX (SERMET) from University of Cantabria were used for sample characterization.

Raman spectroscopy is a light scattering technique, which probes similar low energy vibrational /rotational structures of a molecule. Raman characterisation strategy consisted on excluding the presence of oil neither other contaminant on the collected particles surface. After that, Raman spectroscopy was used to assure that collection surface was free of any contaminants before providing the study of chemical composition and structure of particles. On the other hand, TEM was used to study morphology, particle size and elemental composition. Characterization of morphology with TEM was based on the study of shape of particles and their toxicological risk (Fig. 4).

TEM with EDX showed a mainly spherical morphology. Elemental analysis revealed carbon (C) partially formed by multiwalled carbon nanotubes (Fig. 4) and the presence of Silica (SiO2) and iron oxides.

Raman Spectroscopy detected the presence of disordered graphite. Graphite pentagonal defects let the formation of multiwalled carbon nanotubes, giving flexibility and allowing them to bend. It was also determined the presence of iron oxide nanoparticles in samples (hematite α -Fe2O, Lepidocrocite γ and δ -FeOOH and Siderite FeCO3). The OH vibration of lepidocrocite comes from the presence of water vapour.

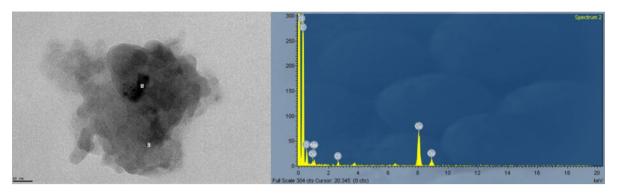


Fig. 5. Image obtained from TEM-EDX and elemental composition visible in its associated spectrum.

Fig. 5 presents an example of a result from TEM-EDX where elemental composition was obtained for two specific points. On the right of the figure a spectrum is also presented. In this particular case, the presence of carbon (C), Sodium (Na), Chlorine (Cl), Cupper (Cu) has been confirmed for a 50 nm nanoparticle.

Conclusions

The proposed method to measure atmospheric nanoparticles charge is a useful approach for the development of multidisciplinary studies where this property can be a key element to expand our knowledge on different fields. For instance, on mechanisms of deposition of organic and inorganic matter inside the human respiratory tract, which can be improve if charge measurement of nanoparticles is considered. Moreover, measurements of charge of atmospheric nanoparticles would allow to elaborate air masses electrical classification, study the links between atmospheric variables and electricity, and examine the links between atmospheric electric fields and wellbeing of living organisms. The sample characterization based on the combination of the indicated spectroscopy techniques provides a useful tactic to better known the structure and composition of atmospheric particulate matter acting as airborne.

Ethics statements

There is no any ethics issue to consider.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Pablo Fdez-Arroyabe: Conceptualization, Methodology, Investigation, Writing – original draft, Writing – review & editing. Ciro Luis Salcines Suárez: Methodology, Data curation, Writing – review & editing. Ana Santurtún: Visualization, Investigation. Ismael Setién: Writing – original draft, Writing – review & editing, Software, Data curation, Validation. Pavlos Kassomenos: Supervision. Tuukka Petäjä: Writing – original draft, Writing – review & editing.

Data availability

The data that has been used is confidential.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mex.2023.102148.

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