



UNIVERSIDADE FEDERAL DE MATO GROSSO
INSTITUTO DE FÍSICA
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LUCAS CARDOSO RAMOS

**Advanced Characterization of Atmospheric Aerosols: Insights into Their
Physicochemical Properties and Atmospheric Processes through Synchrotron-based
Techniques**

CUIABÁ – MT
2026

LUCAS CARDOSO RAMOS

**Advanced Characterization of Atmospheric Aerosols: Insights into Their
Physicochemical Properties and Atmospheric Processes through Synchrotron-based
Techniques**

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Coorientador: Prof. Dr. Flávio César Vicentin

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*“Hoje de manhã atravessando o mar
Vou me perder
Vou me reencontrar
A cada vento que soprar”
- Emicida*

RESUMO

Os aerossóis atmosféricos desempenham papel central na regulação climática, influenciando o balanço radiativo terrestre, a formação de nuvens e a qualidade do ar. Neste trabalho, realizamos uma caracterização multiescala de aerossóis atmosféricos combinando medições *in situ* e análises laboratoriais avançadas, com ênfase em técnicas baseadas em radiação síncrotron. Amostras foram coletadas em biomas-chave da América do Sul, o Pantanal e a Amazônia Central, abrangendo diferentes regimes atmosféricos, desde períodos de intensa queima de biomassa até condições dominadas por emissões biogênicas. A abordagem experimental integrou medidas em tempo real da distribuição de tamanho e composição química molecular de aerossóis (SMPS e ACSM). E métodos de caracterização avançada, como: composição elementar (XRF), química de superfície (XPS), identificação de grupos funcionais por espectroscopia no infravermelho (FTIR) e especiação de grupos funcionais de carbono em nível nanométrico (NEXAFS e STXM). Os resultados revelam uma forte dependência do tamanho de partícula na composição química e no estado de oxidação, com partículas maiores apresentando superfícies mais oxidadas e maior potencial de ativação como núcleos de condensação de nuvens. Observou-se ainda a influência significativa do transporte de longa distância na composição dos aerossóis amazônicos, bem como o aumento de partículas finas, carbono negro e metais traço associados a atividades antrópicas, especialmente durante a estação seca. Os resultados reforçam a importância de abordagens multiescala na investigação de aerossóis em regiões tropicais. A principal contribuição desta tese foi demonstrar que a integração entre observações ambientais e técnicas avançadas baseadas em radiação síncrotron permite acessar informações sobre a composição química e a estrutura dos aerossóis atmosféricos que permanecem inacessíveis às abordagens convencionais. A combinação de medições de campo com análises laboratoriais de alta sensibilidade revelou níveis inéditos de heterogeneidade química e estrutural, evidenciando a complexidade desses sistemas em múltiplas escalas. Os resultados obtidos destacam o potencial transformador das técnicas síncrotron para as ciências atmosféricas, abrindo novas fronteiras para a investigação das propriedades físico-químicas dos aerossóis, seus processos de envelhecimento atmosférico, interações com nuvens e impactos climáticos, com resolução química e espacial sem precedentes.

Palavras-chave: aerossóis atmosféricos; NEXAFS; STXM; Pantanal; Amazônia.

ABSTRACT

Atmospheric aerosols play a central role in climate regulation, influencing the Earth's radiative balance, cloud formation, and air quality. Despite recent advances, significant gaps remain in the understanding of their chemical composition, mixing state, and aging processes, particularly in tropical regions. In this work, we performed a multiscale characterization of atmospheric aerosols by combining in situ measurements and advanced laboratory analyses, with an emphasis on synchrotron radiation-based techniques. Samples were collected in key South American biomes, the Pantanal and the Central Amazon, covering different atmospheric regimes, ranging from periods of intense biomass burning to conditions dominated by biogenic emissions. The experimental approach integrated real-time measurements of aerosol size distribution and molecular chemical composition (SMPS and ACSM), and advanced characterization methods, including elemental composition (XRF), surface chemistry (XPS), identification of functional groups by infrared spectroscopy (FTIR), and carbon functional group speciation at the nanometer scale (NEXAFS and STXM). The results reveal a strong particle-size dependence on chemical composition and oxidation state, with larger particles exhibiting more oxidized surfaces and greater potential for activation as cloud condensation nuclei. A significant influence of long-range transport on the composition of Amazonian aerosols was also observed, as well as an increase in fine particles, black carbon, and trace metals associated with anthropogenic activities, particularly during the dry season. The findings reinforce the importance of multiscale approaches for investigating aerosols in tropical regions. The main contribution of this thesis was to demonstrate that the integration of environmental observations and advanced synchrotron-based techniques enables access to information on the chemical composition and structure of atmospheric aerosols that remains inaccessible through conventional approaches. The combination of field measurements and high-sensitivity laboratory analyses revealed unprecedented levels of chemical and structural heterogeneity, highlighting the complexity of these systems across multiple scales. The results obtained underscore the transformative potential of synchrotron techniques for atmospheric sciences, opening new frontiers for the investigation of aerosol physicochemical properties, aging processes, cloud interactions, and climate impacts, with unprecedented chemical and spatial resolution.

Keywords: aerosols; NEXAFS; STXM; Pantanal; Amazonia

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LIST OF ACRONYMS

ACSM	Aerosol Chemical Speciation Monitor
AE	Ångström Exponent
AERONET	Aerosol Robotic Network
AOD	Aerosol Optical Depth
AR	Accumulated Rain
ATTO	Amazon Tall Tower Observatory
BC	Black Carbon
BCe	Equivalent Black Carbon
BB	Biomass Burning
BLH	Boundary Layer Height
BrC	Brown Carbon
BAPP	Base Avançada de Pesquisa do Pantanal
CAMS	Copernicus Atmosphere Monitoring Service
CAPES	Coordination for the Improvement of Higher Education Personnel
CCN	Cloud Condensation Nuclei
CETESB	Companhia Ambiental do Estado de São Paulo
CNPEM	Centro Nacional de Pesquisa em Energia e Materiais
CO	Carbon Monoxide
CONAMA	Conselho Nacional do Meio Ambiente
CPC	Condensation Particle Counter
DMA	Differential Mobility Analyzer
Dp	Particle Diameter
EDXRF	Energy Dispersive X-ray Fluorescence
Ef	Enrichment Factor
ESS	European Spallation Source
EXAFS	Extended X-ray Absorption Fine Structure
FTIR	Fourier Transform Infrared Spectroscopy
FQ	Focos de Queimada
FRP	Fire Radiative Power
GPD	Global Prediction Data
H:C	Hydrogen-to-Carbon Ratio
HYSPLIT	Hybrid Single-Particle Lagrangian Integrated Trajectory
IC	Inorganic Carbon
IFUSP	Instituto de Física da Universidade de São Paulo
IPCC	Intergovernmental Panel on Climate Change
IR	Infrared Radiation
ITCZ	Intertropical Convergence Zone
LFA	Laboratório de Física Atmosférica
LIDAR	Light Detection and Ranging
MAAP	Multi-Angle Absorption Photometer
MOUDI	Micro-Orifice Uniform Deposit Impactor
NEXAFS	Near Edge X-ray Absorption Fine Structure
NO ₂	Nitrogen Dioxide
NPF	New Particle Formation
OA	Organic Aerosol
O:C	Oxygen-to-Carbon Ratio
O ₃	Ozone
PM	Particulate Matter

PM ₁	Particulate Matter smaller than 1 µm
PM _{2.5}	Particulate Matter smaller than 2.5 µm
PM ₁₀	Particulate Matter smaller than 10 µm
PNHR	Private Natural Heritage Reserve
POA	Primary Organic Aerosol
PTFE	Polytetrafluoroethylene
RH	Relative Humidity
R _n	Net Radiation
ROI	Region of Interest
SE	Standard Error
SESC	Serviço Social do Comércio
Si/Au	Gold-Coated Silicon Wafer
SIA	Secondary Inorganic Aerosol
SMPS	Scanning Mobility Particle Sizer
SOA	Secondary Organic Aerosol
SSA	Single Scattering Albedo
STXM	Scanning Transmission X-ray Microscopy
Ta	Air Temperature
TC	Total Carbon
UFMT	Universidade Federal de Mato Grosso
UV/Vis	Ultraviolet/Visible Spectroscopy
VOC	Volatile Organic Compound
WHO	World Health Organization
XANES	X-ray Absorption Near Edge Structure
XAS	X-ray Absorption Spectroscopy
XPS	X-ray Photoelectron Spectroscopy
XRF	X-ray Fluorescence

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1. INTRODUCTION

Small particles suspended in the atmosphere play a central role in key atmospheric processes, such as the long-range transport of substances, the modification of solar radiation, and adverse effects on human health. In addition, these particles can indirectly influence cloud formation, making clouds brighter or even inducing rainfall in arid regions (Mahilang; Deb; Pervez, 2021). Atmospheric aerosols remain suspended until they are removed by dry or wet deposition and typically range in size from 0.01 to 100 μm (Seinfeld; Pandis, 2006). They originate from diverse sources, including biomass burning, volcanic eruptions, fossil fuel combustion, industrial emissions, sea spray, soil resuspension, and biogenic activity (Alves, 2005). Each emission source imparts unique characteristics to the particles, which can be investigated through size distribution and chemical composition analyses. Moreover, aerosols from different sources differ in their atmospheric evolution and influence on atmospheric processes. Aerosols are defined as primary when emitted directly from their sources, and secondary when formed through subsequent processes, such as reactions with gases or volatile organic compounds (VOCs) (CETESB, 2020).

To clarify the importance of these particles that surround us and are inhaled into our bodies, every year tens of millions of tons of dust from the Sahara Desert cross the Atlantic Ocean via the Intertropical Convergence Zone (ITCZ), supplying essential chemical elements that sustain life in the Amazon rainforest (Artaxo *et al.*, 1998). Aerosols have also inspired humanity throughout history, appearing in the writings of Sir Francis Bacon, the studies of Leonardo da Vinci, and even in the songs of Caetano Veloso (Colbeck; Lazaridis, 2014; Veloso, 1989). Nevertheless, it was only in the last century that aerosol research advanced significantly, driven by scientific and technological progress, and it has now gained increasing prominence within the international community, particularly in the context of climate change alongside greenhouse gases (Colbeck; Lazaridis, 2014). Despite this progress, major knowledge gaps remain, including uncertainties in how aerosols are represented in atmospheric models, what constitutes a safe atmospheric burden of aerosols, the processes governing particle aging, and how their microphysical properties influence both optical behavior and cloud formation (Bzdek; Reid; Cotterell, 2020).

Aerosol characterization is broad and depends on the type of information being sought, by different techniques and equipment it is possible to reach chemical details such as composition and chemical state up to physical properties such as particle size and albedo

(McMurry, 2000). The integration of field measurements and laboratory analyses is widely used to identify emission sources, assess air quality in each region, and investigate the processes of particle formation and aging. In this work, we used advanced synchrotron-based techniques to imaging and measure physicochemical aerosols' properties at nanoscale (~ 100 nm of spatial resolution) and high spectral resolution, aiming on understanding how different aerosols undergo distinct atmospheric processes, how these processes affect particle morphology and chemical state, and what potential impacts they may have on the local atmosphere.

1.1. Aims and hypothesis

The main objective of this study is to employ advanced aerosol characterizations to investigate their physicochemical properties and infer their impact on the atmosphere. To achieve this:

- Collect and prepare suitable aerosol samples from biomes that are critical for climate regulation in South America.
- Measure the physicochemical properties of aerosols both during sampling and in laboratory analysis.
- Characterize aerosols using imaging techniques, obtaining insight of their morphology and chemical information.
- Analyze atmospheric variations in different conditions and their changes to the aerosol's molecular compounds, surface and chemical state.

The central hypothesis of this thesis is that the application of advanced synchrotron-based techniques, in combination with *in-situ* aerosol measurements, enables a level of physicochemical characterization that cannot be achieved by conventional approaches alone. This integrated methodology is expected to reveal previously unresolved aspects of aerosol composition, surface properties, and mixing state, particularly in relation to size-dependent behavior and atmospheric processing. Such insights are essential for advancing the understanding of aerosol formation, aging, and their role in cloud activation and climate interactions in the Central-West and Northern regions of Brazil.

1.2. Thesis structure

In **Chapter 2**, the thesis provides an overview of atmospheric aerosols, covering their sources, formation mechanisms, physicochemical properties, and impacts on cloud formation and climate. In addition, the chapter describes the principal conventional techniques used for aerosol sampling and characterization. It also details the main sampling and characterization techniques employed, with particular emphasis on synchrotron-based tools.

The subsequent chapters present the main achievements of the Ph.D. research. **Chapter 3** investigates the composition of aerosols during the dry season in a wetland area, identifying emission sources, the influence of anthropogenic activities, and the presence of heavy metals. The findings of this chapter raised important questions regarding how human emissions affect the processes of new particle formation. This motivated the research presented in **Chapter 4**, which integrates field measurements, atmospheric modeling, and spectroscopic techniques to analyze the physicochemical properties of size-segregated particles and their environmental impacts. Changes in particle size, molecular group distribution, and surface composition associated with biomass-burning emissions were identified. Building on these results, **Chapter 5** applies advanced imaging and spectroscopic methods to investigate particle morphology, elemental spatial distribution, and carbon functional groups in micro-aggregates, aiming to clarify how particle aging processes influence their physicochemical properties.

Finally, **Chapter 6** highlights how advanced synchrotron-based techniques provide complementary information to conventional aerosol measurements. While traditional methods are effective in determining particle concentration, size distribution, and bulk chemical composition, synchrotron-based analyses allow a more detailed investigation of aerosol particles, revealing how chemical species are distributed within them and how their physicochemical properties vary at the micro and nanoscale. By combining spectroscopic and imaging approaches, this chapter examines the chemical state, elemental distribution, functional groups, and internal heterogeneity of atmospheric aerosols, providing new insights into their physicochemical properties and atmospheric processing.

2. THEORY AND TECHNIQUES

2.1. ATMOSPHERIC AEROSOLS OVERVIEW

The atmosphere is a complex and dynamic system that influences the climate, soil, and human health. Ecosystems are closely linked to local climatic conditions and can be affected by emissions from both regional and distant sources. The upper boundary of the Earth's atmosphere is defined by the Kármán line, located at an altitude of approximately 100 km. Nevertheless, the majority of the atmospheric mass is concentrated at lower altitudes, with about 99% of its total mass contained within the first 30 km (Colbeck; Lazaridis, 2014; Seinfeld; Pandis, 2006). The atmosphere is not homogeneous and may exhibit variations in its composition depending on the region, particularly as a result of local emissions. An important aspect related to climate change is the significant increase in carbon dioxide concentration since the end of the pre-industrial period. Between 1800 and 2005, this concentration rose from 280 to nearly 400 parts per million (ppm), representing an increase of approximately 40% (IPCC, 2021; Reichardt; Timm, 2004). The troposphere is of particular interest in this study because it contains approximately 80% of the total atmospheric mass. Most atmospheric aerosols remain suspended and confined within the boundary layer height (BLH), the maximum altitude reached by tropospheric aerosols. Where they are transported and undergo reactions with other gases and volatile compounds (Pan *et al.*, 2021).

Environmental standards and regulations in Brazil are developed and enforced by CONAMA (Conselho Nacional do Meio Ambiente). According to Resolution No. 491/2018 (CONAMA, 2018), atmospheric pollutants are defined as “any form of matter present in such quantity, concentration, duration, or other characteristics that may render the air unfit or harmful to health, inconvenient to public well-being, damaging to materials, fauna, and flora, or detrimental to safety, property use and enjoyment, or the normal activities of the community.” The CETESB (Companhia Ambiental do Estado de São Paulo) is responsible for developing the Air Quality Report of the State of São Paulo (CETESB, 2020), which serves as a reference for monitoring and includes a group of pollutants known as air quality indicators. Among these, the most notable are particulate matter (PM), carbon monoxide (CO), sulfur dioxide (SO₂), nitrogen dioxide (NO₂), and ozone (O₃). These pollutants are used as parameters because they are frequently detected in the atmosphere and are associated with negative impacts on human health and the environment.

Most international guidelines for maintaining and improving air quality originate from the World Health Organization (WHO), which sets global targets to keep PM concentrations within levels considered safe for human health. In 2021, the WHO established that the 24-hour mean concentration of PM_{2.5} should be below 15 $\mu\text{g m}^{-3}$, while the corresponding limit for PM₁₀ should be below 45 $\mu\text{g m}^{-3}$, ensuring good air quality (World Health Organization, 2021). In Brazil, the CETESB, through its Air Quality Report, defines reference thresholds to identify episodes of acute air pollution, based on particulate matter concentrations collected on filters exposed to the atmosphere for 24 hours. According to the report, for PM_{2.5}, a concentration of 125 $\mu\text{g.m}^{-3}$ represents an alert condition, while 250 $\mu\text{g.m}^{-3}$ indicates an emergency condition. For PM₁₀, the corresponding thresholds are 250 $\mu\text{g m}^{-3}$ (alert) and 500 $\mu\text{g m}^{-3}$ (emergency) (CETESB, 2020).

Despite the considerable progress in understanding atmospheric aerosols, significant knowledge gaps remain regarding their formation and aging mechanisms, as well as how their microphysical properties determine macroscopic optical behavior. Moreover, the influence of aerosols on cloud formation and the environmental and human health impacts associated with high concentrations of particulate matter (PM) are still not fully understood (Alves, 2005; Bzdek; Reid; Cotterell, 2020; Richardson *et al.*, 2023; Seinfeld; Pandis, 2006).

2.1.1. Aerosols formation and composition

Atmospheric aerosols play fundamental roles in the atmosphere. They consist of a mixture of particles with diameters ranging from 0.01 to 100 μm , which remain suspended in the air until they are removed by precipitation or by other deposition mechanisms, such as interaction with vegetation or surface settling (Seinfeld; Pandis, 2006; Zhang, Leiming *et al.*, 2001). The types of aerosols are primarily determined by their emission sources (Fig. 1), which can be either anthropogenic or natural. Natural sources include soil dust, sea salt, volcanic emissions, and biogenic particles, while anthropogenic emissions originate mainly from biomass burning, fossil fuel combustion, and vehicular and industrial activities. Particles released from human activities generally have diameters smaller than 2.5 μm and are known to cause harm to both the environment and human health. Previous studies have linked the increase in respiratory diseases and viral transmission to PM concentrations and environmental

parameters (Brunelli *et al.*, 2021; Nardocci *et al.*, 2013; Setti *et al.*, 2020). Aerosol particles are typically classified according to their aerodynamic diameter into distinct size modes.

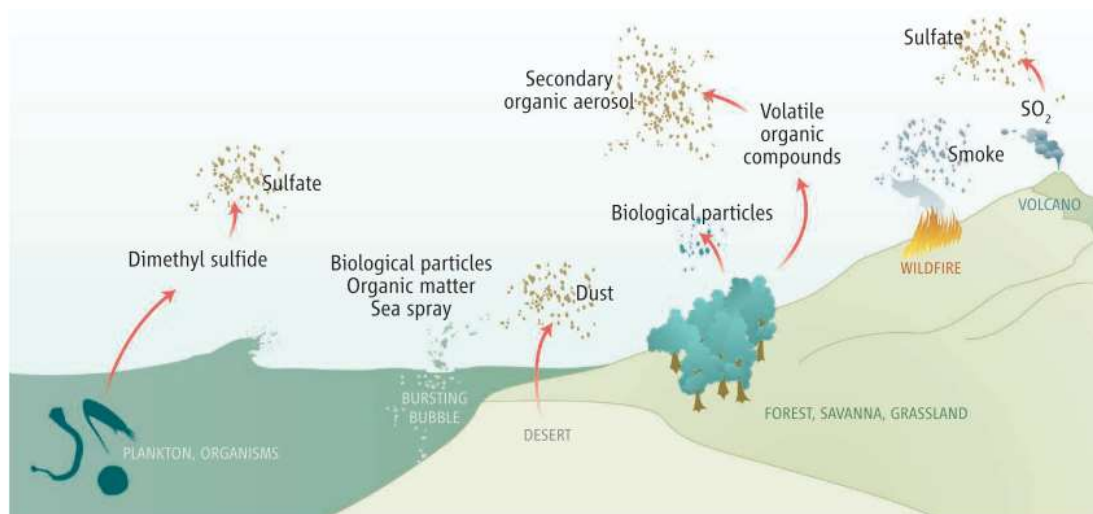


Figure 1. Schematic representation of different primary and secondary sources of atmospheric aerosols (Meinrat O. Andreae, 2007)

Primary aerosols are those emitted directly from a source, such as Primary Organic Aerosols (POA). In contrast, secondary aerosols are formed in the atmosphere through chemical reactions between pollutants and/or natural constituents. They depend on gaseous precursors and are produced by coagulation or gas-to-particle conversion processes, as in the case of Secondary Organic Aerosols (SOA), which originate from volatile organic compounds (VOCs). Among secondary aerosols, there is also the Secondary Inorganic Aerosol (SIA). Recent studies have revealed that these different types of aerosols can coexist within the same particle, making our understanding of aerosol systems even more complex (Huang *et al.*, 2021a).

One of the key roles of aerosols in atmospheric dynamics is the long-range transport of substances across the planet. This ability arises from the porous structure of particle aggregates, their high surface tension, and the chemical composition determined by the emission process, combined with the strong temporal and spatial variability of aerosols in the atmosphere. Moreover, the formation of secondary products, such as organic acids derived from volatile organic precursors, is also a plausible and significant process in atmospheric chemistry (Santos, Djacinto Monteiro Dos *et al.*, 2021).

Particle size is a key parameter associated with new particle formation (NPF) processes (Colbeck and Lazaridis, 2014). Recent studies on NPF in the upper troposphere over the Amazon (Shen et al., 2024) have demonstrated the importance of this unique atmospheric process in the region. The nucleation mode is linked to particles that range lower than 7 nm, while, Aitken modes range from 7 to 100 nm, comprise ultrafine aerosols mainly formed through gas-phase nucleation and condensational growth (Aliaga *et al.*, 2025). The accumulation mode, ranging from about 100 to 440 nm, consists of particles produced by the coagulation of smaller particles and the accumulation of condensable vapors, as shown in Figure 2. Finally, the coarse mode, including particles with diameters larger than 1 μm , is primarily associated with mechanical processes such as dust resuspension, sea spray, and biogenic (Colbeck; Lazaridis, 2014; Seinfeld; Pandis, 2006).

Size range [nm]	Notation	Description
2–4	$N_{2-4}^{-(+)}$	– Primarily observes reliable negative (positive) ion concentrations – Indicates early clustering and growth – Marks the onset of nucleation
4–7	N_{4-7}	– Falls within the lower nucleation range – Sensitive to the intensity of new particle formation (NPF) – Marks early nucleation and growth stages
7–13	N_{7-13}	– Represents the upper nucleation range – Reflects further growth – Not influenced by primary emissions at EAC during morning and afternoon rush hours
13–40	N_{13-40}	– Corresponds to the lower Aitken mode – Most NPF-produced particles are found in this range by the end of daily NPF events – Captures traffic primary emissions at EAC
40–100	N_{40-100}	– Pertains to the upper Aitken mode – A small but significant portion of NPF-produced particles reach this size – Sensitive to traffic primary emissions at EAC
100–440	$N_{100-440}$	– Denotes the accumulation mode – Affected by primary emissions from pollution and dust – Notably influenced by volcanic plume influences in this specific geographic area

Figure 2. Different particle size ranges for nucleation ($D_p < 7$ nm), Aitken mode ($D_p < 100$ nm), and accumulation mode ($D_p > 100$ nm), and a description of their characteristics (Aliaga et al., 2025)

Given the different types of carbonaceous components that constitute aerosols, it is essential to standardize their nomenclature by considering both the physicochemical properties of the particles and the measurement techniques employed (Petzold *et al.*, 2013). Accordingly, the various carbonaceous species can be defined as follows: Total Carbon (TC) refers to the total mass of all carbonaceous material contained in the aerosol, encompassing both organic and

inorganic forms of carbon. Inorganic Carbon (IC) corresponds mainly to inorganic carbonate salts that can be present as minor constituents of atmospheric particles. Among the carbonaceous species, BC is distinguished by several specific characteristics. It strongly absorbs visible light, particularly around a wavelength of 550 nm for freshly emitted particles. BC is a refractory substance, exhibiting a volatilization temperature close to 4000 K, and is insoluble in both water and organic solvents such as methanol and acetone, as well as in other components of atmospheric aerosols. Structurally, BC consists of aggregates of small carbon spherules with diameters ranging from less than 10 nm up to approximately 50 nm. Its composition is dominated by graphitic carbon atoms with sp^2 hybridization, giving rise to its high optical absorption and chemical stability. In contrast, Brown Carbon (BrC) represents the light-absorbing organic fraction of atmospheric aerosols and originates from a variety of natural and anthropogenic sources, including humic-like substances from soils, combustion tar materials, and biogenic emissions. BrC is characterized by a brownish appearance, which results from its non-uniform absorption across the visible spectrum, its light absorption increases as the wavelength decreases toward the blue region of visible light. When emitted primarily from biomass burning, BrC typically contains molecular markers such as nitroaromatic compounds and polyphenols. In contrast, secondary BrC formed through atmospheric chemical reactions can be identified by the presence of markers such as methyl-nitrocatechol (Yan *et al.*, 2018).

Carbon, in its various chemical forms, is a key constituent of atmospheric aerosols. Its origin may be associated with biogenic emissions, the formation of secondary products through atmospheric chemical reactions, or biomass burning events (Ramos *et al.*, 2024; Vieira *et al.*, 2023). Carbonaceous aerosol particles are predominantly associated with the fine particulate matter fraction. In tropical regions, this type of aerosol can account for up to 90% of the total fine particle mass, whereas in mid-latitude regions, its contribution typically ranges between 20% and 80% (Monks *et al.*, 2009). As individual carbon particles, carbon exhibits strong light-absorbing properties, particularly at wavelengths between 514.5 nm and 633 nm. In its graphite form, carbon atoms possess three sp^2 -hybridized valence electrons, formed by the combination of 1 orbital and 2 p orbitals, while the fourth valence electron remains perpendicular to the plane in a π orbital that does not participate in covalent bonding and is only weakly held (Bond, Tami C.; Bergstrom, 2006).

The comparison between oxygen and carbon proportions (O/C ratio) is an important tool for identifying structural characteristics and the nature of carbon species in aerosol samples.

This ratio depends on both the source of the material and the combustion conditions to which it has been exposed. Samples with a high content of elemental carbon typically exhibit a low O/C ratio, indicating a lower degree of oxidation (Brodowski *et al.*, 2005; Reid *et al.*, 1998). The O/C ratio is also associated with key morphological characteristics of Black Carbon (BC), influencing parameters such as particle size, shape, and surface texture, which may be rough, irregular, smooth, or exhibit a non-charred or charred core. It also provides insights into the particle type (residual or soot) and its likely emission source, such as biomass burning, fossil fuel combustion, or coal combustion. Furthermore, aerosols composed of POA, SOA, and SIA can exhibit up to three distinct liquid phases under relative humidity conditions ranging from 90% to 20%, provided that the SOA has an O/C ratio below 0.43. However, when this ratio exceeds 0.8, the formation of three distinct phases does not occur (Huang *et al.*, 2021a).

2.1.2. Direct and indirect influences of aerosols on climate

Aerosols exert a direct effect on solar radiation, either by scattering or absorbing light, depending on their chemical composition and physical properties. Aerosols that predominantly scatter radiation tend to contribute to atmospheric cooling, whereas those that absorb radiation promote warming. These direct effects can lead to significant local climate alterations and impact regional meteorological processes. Aerosol concentrations also exhibit seasonal variability, with markedly higher levels during dry periods compared to wet seasons, reflecting changes in emission sources and atmospheric dynamics (Morais, Fernando G. *et al.*, 2022; Palácios *et al.*, 2022). Aerosol composition is a key factor influencing the direct radiative effect. Sulfate-rich aerosols predominantly scatter incoming solar radiation, thereby contributing to a cooling effect. In contrast, aerosols containing black carbon (BC) strongly absorb solar radiation, leading to atmospheric heating and a positive radiative forcing (IPCC, 2021; Seinfeld; Pandis, 2006).

Another key property of these intriguing atmospheric agents is their hygroscopicity, which refers to the ability of aerosol particles to absorb water vapor from the atmosphere as a function of ambient humidity and water vapor pressure. This property enables aerosols to act as cloud condensation nuclei (CCN), influencing the formation and intensity of precipitation in a given region (Rizzo, Luciana Varanda; Artaxo, 2002). The term “indirect effects” of aerosols refers to their influence on radiation through modifications in cloud microphysical properties,

rather than through direct interactions with radiation. These effects can lead to significant changes in cloud characteristics, potentially impacting local climate and surface radiation. Aerosol–cloud interactions remain one of the largest sources of uncertainty in the climate system (IPCC, 2021).

2.1.3. Atmospheric circulation and climate change

Atmospheric aerosols exhibit strong temporal and spatial variability, meaning that after emission they can be transported over long distances across the globe. Intercontinental air mass transport between Africa and the Amazon Basin is also considered an important source of aerosols, including mineral dust and biomass burning products, contributing to the composition and variability of atmospheric particles over the Amazon region (Holanda *et al.*, 2020). Atmospheric circulation plays a fundamental role in the transport and redistribution of aerosols on regional and global scales. Wind patterns at different levels of the troposphere, associated with systems such as the Intertropical Convergence Zone (ITCZ) and large-scale circulation cells, control the movement of air masses and, consequently, the flux of particles between continents and oceans (Artaxo *et al.*, 2022; Pullen *et al.*, 2017). In addition to horizontal transport, vertical atmospheric motions such as convection and subsidence influence the vertical distribution of aerosols and their interaction with clouds (Andreae, Meinrat O. *et al.*, 2018). Therefore, atmospheric circulation not only determines the origin and fate of particles but also modulates aerosol formation, growth, and chemical transformation processes in the atmosphere.

Atmospheric processing begins shortly after emission, and as particles age through chemical reactions with gases and interactions with other particles, their physicochemical properties are modified (Lee *et al.*, 2020). These transformations directly influence atmospheric properties, impacting climate at both local and global scales. Climate change lies at the core of global environmental debates and remains a challenging topic due to the significant uncertainties associated with its prediction and quantification (Richardson *et al.*, 2023). The uncertainties in the climate models arise from the interplay of multiple processes operating across different spatial and temporal scales, including atmospheric dynamics, land–atmosphere interactions, and biogeochemical cycles (Bzdek; Reid; Cotterell, 2020; Doherty; O’Connor; Turnock, 2022; Vu; Hoang; Nguyen, 2023).

2.1.4. Real-time aerosol measurements

There are many ways to measure atmospheric particles and their physicochemical characteristics through optical measurements while they remain suspended in the atmosphere (McMurry, 2000). Among the remote sensing techniques used for atmospheric aerosol monitoring, the Aerosol Robotic Network (AERONET) is one of the most widely employed worldwide. AERONET consists of a global network of ground-based sun photometers that continuously measure solar radiation at multiple wavelengths, typically ranging from 340 to 1640 nm (Holben et al., 1998). Measurements are usually acquired every 15 minutes under cloud-free conditions and are used to retrieve a variety of aerosol optical properties.

The primary product derived from AERONET observations is the Aerosol Optical Depth (AOD), which quantifies the extinction of solar radiation caused by scattering and absorption by atmospheric particles along the atmospheric column at same wavelength (Brunelli, 2020). Inversion algorithms provide several important aerosol parameters, including the Ångström Exponent (AE), which is related to particle size. The Single Scattering Albedo (SSA), which describes the relative contribution of scattering and absorption. In addition, it provides the volume particle size distribution, the complex refractive index, and the fine-mode fraction. Together, these properties provide valuable information about aerosol loading, particle size, chemical composition, and potential emission sources (Morais, Fernando G. *et al.*, 2022; Palácios *et al.*, 2022; Zhang, Xindan *et al.*, 2022). Due to its long-term observations and global coverage, AERONET has become one of the most important datasets for aerosol research, supporting studies of aerosol transport, source attribution, aerosol–cloud interactions, and climate impacts.

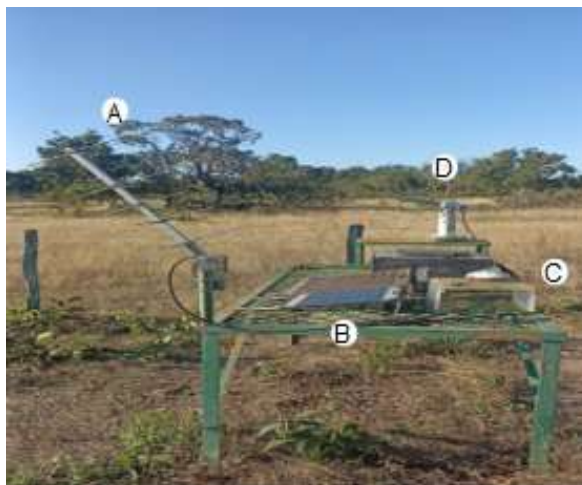


Figure 3. AERONET station MIRANDA - CUIABÁ located at Fazenda Miranda. (A) Data transmission system; (B) solar panel and power supply system; (C) pyranometer; and (D) collimator of the AERONET robotic sun photometer.

Atmospheric aerosols exhibit a wide range of chemical species in their composition, as well as significant spatial and temporal variability. Therefore, real-time measurements of their physicochemical properties are of great scientific interest. Particle size is one of the main indicators of aerosol evolution. Ultrafine particles with diameters (D_p) smaller than 30 nm are typically associated with nucleation, those between $30 < D_p < 100$ nm with coagulation, and particles in the range of $100 < D_p < 500$ nm with aggregation. Each of these processes modifies the physical and chemical properties of the particles, thereby influencing the atmosphere in distinct ways (Colbeck; Lazaridis, 2014; Fan *et al.*, 2021; Franco *et al.*, 2022).

The Scanning Mobility Particle Sizer (SMPS), showed in Figure 4, can measure particle size distributions ranging from approximately 0.25 to 1000 nm. Aerosols drawn into the instrument are electrically charged using a neutralizer and then introduced into a differential mobility analyzer (DMA), where a radial electric field separates particles according to their electrical mobility, which is directly related to particle size. The particles are then counted, allowing the determination of the distribution of size. The data obtained are essential for studies on particle growth, cloud condensation nuclei (CCN) formation, and dew point efficiency (Artaxo *et al.*, 2022; Mahilang; Deb; Pervez, 2021; Mcmurry, 2000; Pöhlker, Mira L. *et al.*, 2018).



Figure 4. Electrostatic aerosol particles are charge-neutralized, size-classified by electrical mobility in a DMA, and counted by a Condensation Particle Counter (CPC), allowing the measurement of particle number size distributions (Inc., 2026)

The composition of atmospheric aerosols has a major influence on atmospheric dynamics, affecting both the radiative balance and cloud formation processes. These effects depend strongly on the local aerosol population circulating in the atmosphere. The formation and aging processes of aerosols are also influenced by secondary organic aerosols (SOA), which can be quantified using the Aerosol Chemical Speciation Monitor (ACSM) (Hu *et al.*, 2015; Meller *et al.*, 2025). The ACSM is a mass spectrometer designed for continuous measurements of the chemical composition of non-refractory aerosols, i.e., particles that vaporize at moderate temperatures. Consequently, the instrument does not detect refractory components such as silicates, soot, or metals. Before entering the detection chamber, the aerosol flow is passed through a dryer to remove water vapor and minimize interference. The dried particles are then focused by an aerodynamic lens onto a heated surface, where they are vaporized. The resulting gases are ionized and analyzed by mass spectrometry. These measurements provide real-time information on the concentrations and chemical composition of key species such as nitrate, sulfate, ammonium, and organics (McMurry, 2000; Ponczek *et al.*, 2021b).

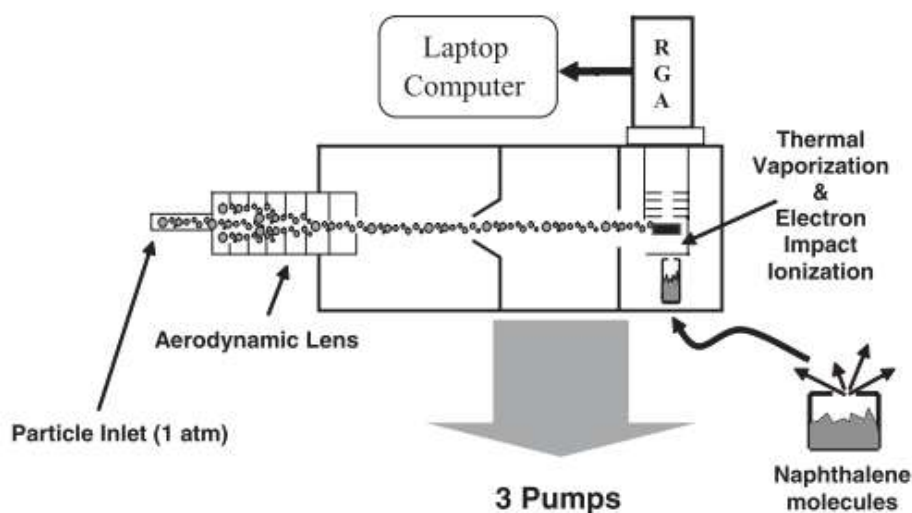


Figure 5. Schematic representation of Aerosol particles are focused into a beam, vaporized, ionized by electron impact, and analyzed by mass spectrometry to determine the chemical composition of non-refractory submicron aerosols (Ng *et al.*, 2011)

Overall, the combination of remote sensing and *in-situ* observations provides a powerful framework for investigating atmospheric aerosols across multiple spatial and temporal scales. While remote sensing offers continuous large-scale monitoring, direct measurements are essential for validating observations, reducing uncertainties, and characterizing aerosol physicochemical properties in greater detail. Together, these approaches contribute to a more complete understanding of aerosol sources, evolution, and climatic effects. A comprehensive overview of current measurement techniques and their applications is provided by (McMurry, 2000). Establishing this foundation is essential for integrating conventional aerosol observations with advanced synchrotron-based characterization techniques, enabling the investigation of aerosol physicochemical properties at an unprecedented level of detail.

2.2. RADIATION AND AEROSOLS INTERACTIONS

The interactions between aerosols and radiation are important for two main reasons. The first is the emergence of aerosol optical properties, which directly influence the atmosphere and the net radiation balance reaching the Earth's surface. When exposed to solar radiation, aerosols scatter and absorb light, producing visible effects such as reduced visibility and changes in sky coloration, often appearing more reddish, especially during sunrise or sunset, depending on

particle concentration and size. These optical properties are strongly influenced by the microphysical characteristics of aerosols, although this relationship remains not fully understood (Bzdek; Reid; Cotterell, 2020).

The second reason why the interaction between radiation and matter is important is that it enables a deeper investigation of aerosols, allowing inferences about their type, size and shape, concentration, chemical composition, and emission sources. In this context, there is a wide range of methods for aerosol detection, which can be broadly categorized into three groups: in situ measurements, remote sensing, and laboratory analyses. In situ measurements employ instruments such as nephelometers and aethalometers to quantify light scattering and absorption by particles. Laboratory analyses involve more direct interactions, including techniques such as UV/Vis spectroscopy, Raman spectroscopy, and X-ray spectroscopy, which provide extensive possibilities for investigating aerosol composition (Ault; Axson, 2017).

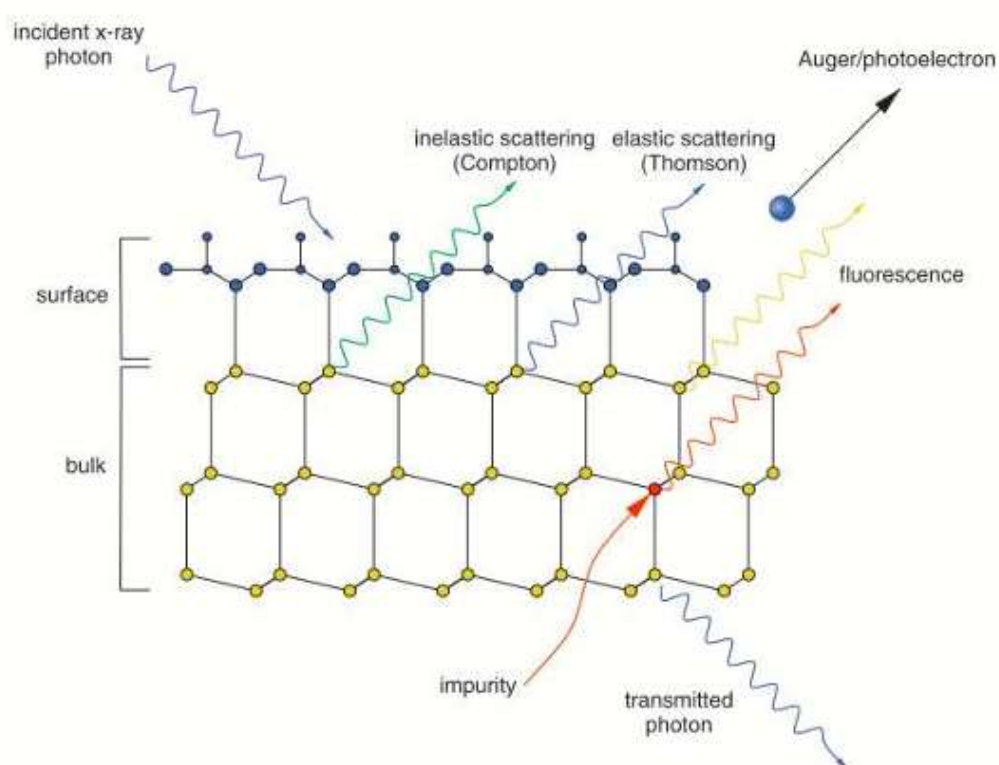


Figure 6. Interaction between X-rays and matter. Physical and structural properties may exhibit distinct characteristics between the surface and the bulk of a material (Willmott, 2011)

Finally, to understand better the nature of the interactions between radiation and aerosols, we need to keep in mind two main criteria to classify them, according to Stephens (2001):

- The **type of target**, that is, whether the radiation interacts with electrons, atoms, or nuclei.
- The **type of event** resulting from the interaction, such as absorption, scattering, or pair production.

2.2.1. Infrared Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy is a versatile technique for characterizing organic and inorganic functional groups present in a sample. Molecules absorb infrared radiation within the spectral range of $4000\text{--}400\text{ cm}^{-1}$, which excites their vibrational modes. These vibrations are primarily classified into two types: stretching and bending modes. A single molecule may exhibit multiple active vibrational modes. For example, the H_2O molecule presents a symmetric stretching mode at approximately $\sim 3657\text{ cm}^{-1}$ and a bending mode around $\sim 1595\text{ cm}^{-1}$. These result in absorption features at different spectral bands that together confirm the presence of water. In more detailed analyses, stretching vibrations are typically associated with the identification of specific chemical bonds, whereas bending modes can provide insights into molecular geometry and structural configuration (Coates, 2000). Figure 7 schematically represents the interaction of the infrared (IR) beam with the sample, highlighting the excitation of vibrational modes and illustrating the corresponding vibrational motions.

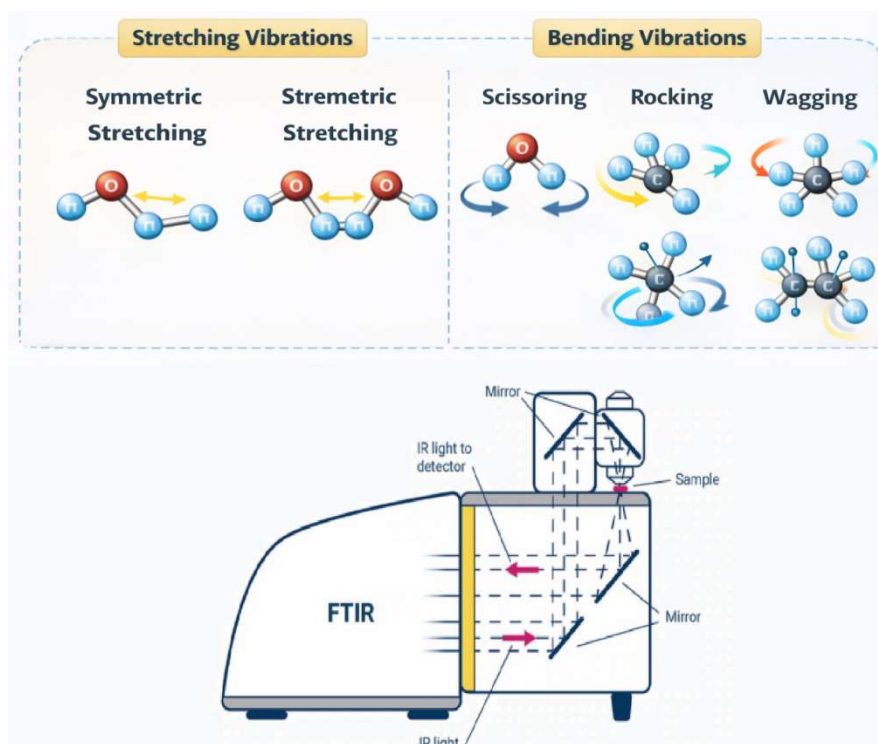


Figure 7. Schematic representation of the FTIR technique. An incident infrared beam excites molecular vibrational modes, and the different motions of the molecules generate absorbance peaks that reflect both the molecular identity and the type of vibration (Agilent, 2025)

Infrared spectroscopy is a versatile technique for the analysis of atmospheric aerosols, as it requires relatively simple sample preparation, causes minimal sample damage, does not require vacuum conditions, and provides valuable information on emission sources, aging processes, and the chemical properties of particulate matter (PM). Several functional groups can be identified using FTIR, including carbonyl ($\text{C}=\text{O}$), carbon-carbon double bonds ($\text{C}=\text{C}$), carboxylate ions (COO^-) (Rabha; Saikia, 2019), organic nitrates ($-\text{ONO}_2$), C-O bonds, ammonium (NH_4^+), and sulfate (SO_4^{2-}) (Coury; Dillner, 2009; Popovicheva; Ivanov; Vojtisek, 2020)

2.2.2. Synchrotron Light

High-standard scientific laboratories, such as those operating electron accelerators (Figure 8), produce radiation of exceptionally high brightness, coherence, and intensity. In these systems, electrons are accelerated to velocities close to the speed of light and, when their trajectories are perturbed by magnetic fields, they emit electromagnetic radiation with characteristic energies, known as synchrotron light.

The generation and control of this light depend on a series of experimental conditions and specialized components, including vacuum environments, undulators, wigglers, magnetic dipoles, mirrors, slits, and cooling systems, which guide the beam to the experimental stations. At these stations, it is possible to perform nanoscopic-scale measurements with high chemical and structural resolution. The use of this light source, together with the diversity of analytical techniques it enables, provides a powerful means to address complex questions in the field of atmospheric aerosols, particularly regarding their chemical composition, structure, and formation processes (Rabha; Saikia, 2019; Willmott, 2011). Recent advances in synchrotron-based techniques have opened a new frontier in scientific research, enabling the measurement of physicochemical properties at spatial and chemical resolutions previously unimaginable. Unlike conventional field measurements, which typically provide bulk or population-averaged information, these techniques allow direct investigation of surface chemistry, mixing state, elemental distribution, and chemical speciation at the level of individual particles and even sub-particle domains. As a result, they provide access to previously inaccessible information, offering new opportunities to understand aerosol formation, aging, and their interactions with the atmosphere.



Figure 8. Synchrotron facilities utilized for advanced aerosol characterization. On the top is Sirius, the Brazilian synchrotron light source located in Campinas, Brazil. The picture on the bottom is MAX IV Laboratory, located in Lund, Sweden (LU, 2025; MCTI, 2025)

The nature of electromagnetic radiation allows light to behave like a quantized energy particle, called a photon. In general, the energy of a photon can be expressed as (Willmott, 2011):

$$E_{\text{photon}} = h\nu = hc/\lambda \quad (1)$$

The Planck's constant ($h = 6.626 \times 10^{-34} \text{ J}\cdot\text{s}$), c the speed of light in vacuum, and λ the wavelength of light in vacuum.

The infrared (IR) region is approximately between $3 \times 10^{12} \text{ Hz}$ and $4.3 \times 10^{14} \text{ Hz}$. This energy range can interact with molecules at the molecular level, inducing characteristic vibrational modes, such as stretching and bending vibrations. IR spectroscopy is widely used for the chemical characterization of aerosols, as it allows the identification of functional groups

based on the specific vibrational frequencies of chemical bonds. However, conventional instruments have limitations in analyzing individual particles larger than approximately $5\text{ }\mu\text{m}$ (Ault; Axson, 2017). The X-ray (X) spectrum, in contrast, is characterized by high frequencies ($3 \times 10^{16} - 3 \times 10^{19}\text{ Hz}$, corresponding to $10^2 - 10^5\text{ eV}$ of energy). As a result, it has very short wavelengths, on the order of micrometers to nanometers, allowing for targeted measurements and high spatial resolution within the sample (Signorelli; Reid, 2011).

To generate synchrotron radiation, an electron beam is accelerated to velocities close to the speed of light and, when its trajectory is deflected by magnetic fields, it emits high-intensity, highly bright electromagnetic radiation, known as synchrotron light. This radiation spans a wide energy range of the electromagnetic spectrum. Subsequently, light is subjected to optical selection and focusing processes, which allow the production of a monochromatic and highly collimated beam, capable of reaching nanoscopic dimensions (Figure 9).

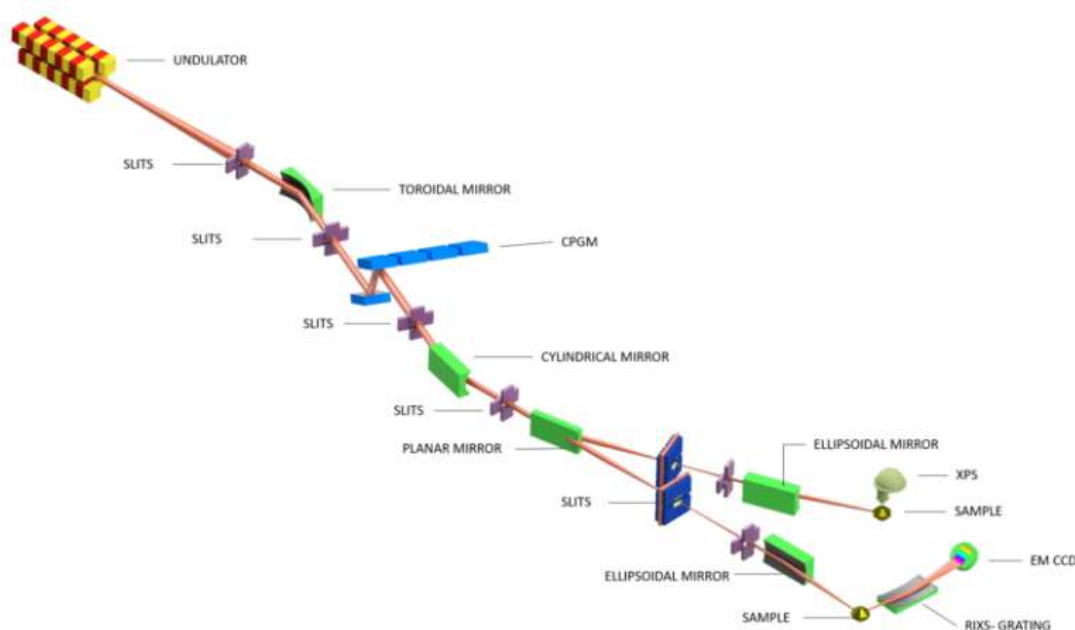


Figure 9. Technical schematic of the IPÊ beamline. Synchrotron radiation is generated by an undulator source, then aligned, conditioned, and focused by the beamline optical system before being delivered to the experimental stations for sample analysis

Beamlines are facilities that integrate a wide range of scientific and technological expertise to create the conditions required for spectroscopy, absorption, scattering, and

microscopy experiments. The high-quality data provided by synchrotron-based experiments enable studies at the frontiers of knowledge across various scientific disciplines. Radiation beam reaches the **endstations** (Figure 10), and it starts the interaction with the samples, producing information about the processes occurring during this interaction. This allows the application of a variety of analytical techniques to characterize the material. In this work, aerosol samples were used to analyze different characteristics of these particles, including concentration, chemical composition, elemental speciation, and morphology.

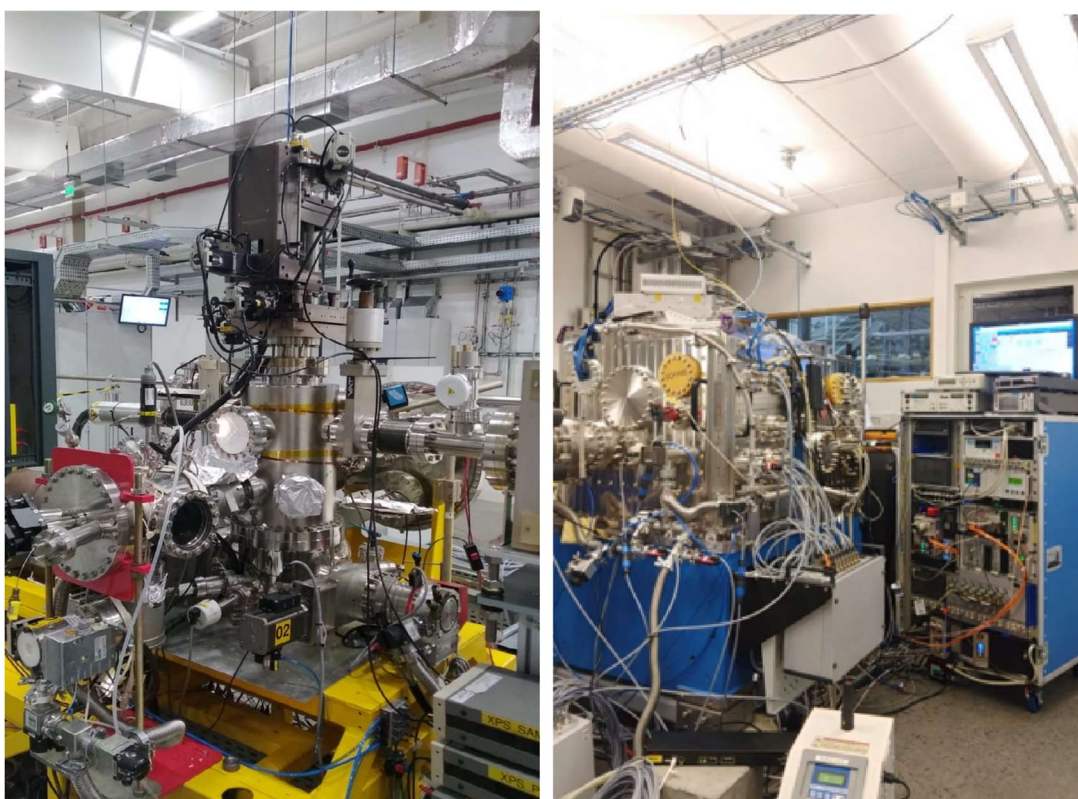


Figure 10. Experimental endstations used for synchrotron-based environmental aerosol characterization. The IPÉ Endstation (left) at Sirius and the SHOPIE Endstation (right) at MAX IV Laboratory. These facilities are dedicated sample environments where materials are analyzed using synchrotron radiation, enabling advanced physicochemical characterization with high sensitivity and spatial resolution.

2.2.3. X-ray Absorption and Scanning Transmission X-ray Microscopy

X-ray absorption occurs when electromagnetic radiation interacts with the electrons of an atom, transferring enough energy to excite or even eject them. When the excitation energy (E_{exc}) matches the resonance energy of an atomic electron, the spectrum exhibits an absorption edge characteristic of that element. The energy range associated with a given element generally includes three regions: pre-edge, edge, and post-edge, each providing distinct spectral features.

The analysis of the edge region is referred to as X-ray Absorption Near Edge Structure (XANES), or, when using soft X-rays, Near Edge X-ray Absorption Fine Structure (NEXAFS). In contrast, the post-edge region is studied through Extended X-ray Absorption Fine Structure (EXAFS). Investigating the absorption edge of a specific element enables the study of electronic transitions and bonding environments. The term *speciation* is used to describe the identification of chemical species within a sample based on the position and shape of absorption peaks (Hitchcock, 2015).

Aerosols exhibit intrinsic optical properties, among which transmittance represents the fraction of light transmitted through the material, defined as the ratio between the transmitted intensity and the incident light intensity (Daemme *et al.*, 2021). Absorbance, in turn, quantifies the amount of light energy absorbed by the material. The Beer–Lambert law is essential for the quantitative understanding of the interaction between radiation and matter, as it relates absorbance to the molar concentration of the absorbing species, the molar extinction coefficient, and the optical path length of the light (Braga, Mauro Sergio *et al.*, 2019), such that:

$$A = \epsilon L c \quad (2)$$

Therefore, transmittance (T) and absorbance (A) are inversely related quantities and can be mathematically expressed, respectively, as (Hardesty; Attali, 2010):

$$T = \frac{I}{I_0} \quad (3)$$

$$A = OD = -\log_{10} \frac{I}{I_0} \quad (4)$$

Where OD is the Optical Density of some element, I_0 is the incident radiation and I the transmitted radiation.

The experimental setup for Scanning Transmission X-ray Microscopy (STXM) includes the fundamental components of a synchrotron beamline, such as an undulator, precision slits, and optical elements. Since STXM applied to light elements operates in the soft X-ray range (100–2500 eV), the measurements must be conducted under high vacuum conditions ($<10^{-6}$ mbar). Additionally, because STXM is an imaging technique, the sample stage must allow nanometric precision movements, typically achieved with piezoelectric motors for fine adjustments. A key component of the setup is the zone plate, which focuses the beam to nanometric dimensions at the sample plane and can be fabricated from different materials. The

detector used in this technique is usually a photomultiplier tube (PMT), a photosensitive cathode that records the intensity of photons transmitted through the sample, as shown in Figure 11.

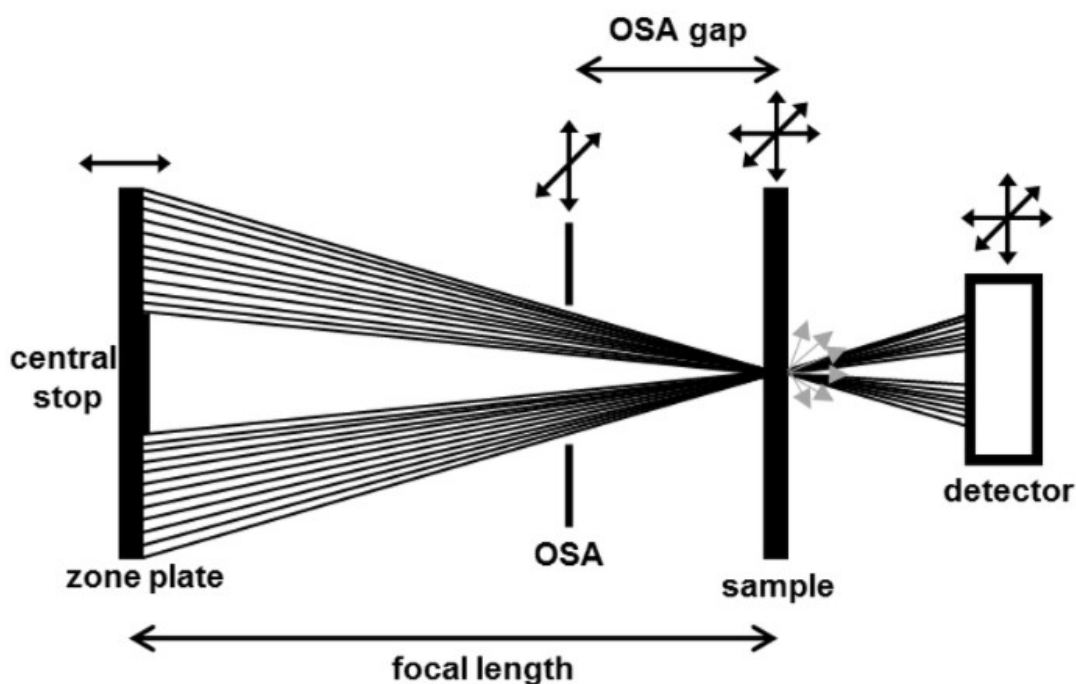


Figure 11. Representative schematic of Scanning Transmission X-ray Microscopy (STXM). Monochromatic soft X-rays are focused by a zone plate and pass through the Optical Selecting Aperture (OSA), which reduces unwanted diffraction and helps focus the beam onto. Detection system is based on a photomultiplier tube (Späth, 2019).

One of the main challenges of STXM at low energy is sample preparation, as the specimens must be sufficiently thin to allow X-ray transmission. For edges such as carbon (~284 eV), sample thicknesses of approximately 100 nm are required to ensure reliable quantification. Measurements of C K-edge are specially challenging, the conditions of alignment and vacuum must be very delimited thus their low cross section, i.e. probably of interaction between X-ray and C K-edge is low. Each image is acquired at a fixed energy, which means that data acquisition is inherently three-dimensional: the X and Y dimensions correspond to the 2D spatial imaging, while the Z dimension corresponds to the energy stack. Prior to data collection, it is necessary to define the region of interest (ROI), the spatial resolution (set by the X and Y step sizes), and the spectral resolution (set by the energy step in Z). These parameters are critical to balance data quality with acquisition time. For optimal results, stacks for each

element should be cleaned to remove noisy or corrupted images, and all images must be properly aligned before analysis.

Given the large variety of carbonaceous compounds present in aerosols, STXM is a powerful tool to investigate the distribution of carbon functional groups in atmospheric particles. The technique provides high-quality analytical data that can clarify the atmospheric processes experienced by aerosols during aging, and how these processes modify their morphology and chemical state (Hopkins *et al.*, 2007; Moffet *et al.*, 2016).

2.2.4. X-ray Fluorescence and Nanoscale Scanning X-ray Fluorescence Microscopy

X-ray fluorescence (XRF) emissions occur after an atom absorbs energy from an incident X-ray or from high-energy electrons, these emissions are driven by the relaxation of excited atoms. When energy is absorbed, core electrons are ejected from the atomic shell, creating an energy vacancy. To restore stability, another electron within the atom transitions to a lower-energy state, releasing part of its excess energy as electromagnetic radiation. These emissions are characteristic of specific electronic transitions and, more importantly, of each chemical element. For this reason, XRF signals provide a powerful analytical tool for the identification and quantification of chemical elements. In practice, all elements in the sample whose absorption energy is lower than the excitation energy of the beam ($E_{\text{binding}} < E_{\text{exc}}$) will emit fluorescence and can be detected. The energy difference between two distinct levels is well defined and depends on the chemical element and the type of electronic transition involved. However, there is an intrinsic uncertainty associated with this energy difference, which arises from the Heisenberg Uncertainty Principle, $\Delta E \Delta t \sim \hbar$, establishing a fundamental limit to the simultaneous determination of the energy and the lifetime of the excited state. Nevertheless, the characteristic energies can typically be measured with a precision of approximately 0.1 eV (Kopittke *et al.*, 2018; Willmott, 2011).

Moseley's law describes the systematic relationship between the characteristic radiation of the most intense spectral lines and the atomic number (Z) of the element, expressed as (Willmott, 2011):

$$\nu = K (Z-1)^2 \quad (5)$$

In the equation, $K = 2.47 \times 10^{15}$ Hz. Moseley's law is based on the Bohr atomic model, according to which the spectral lines have energies corresponding to the differences between discrete orbitals.

The nomenclature of X-ray fluorescence (XRF) is somewhat complex, as it involves transitions between two electronic orbitals. The K, L, and M shells account for the main characteristic emissions. The work by Thompson *et al.* (2001) provides the characteristic emission values for elements with $3 \leq Z \leq 95$, considering the strongest lines: $K\alpha_1$, $K\alpha_2$, $K\beta_1$, $L\alpha_1$, $L\alpha_2$, $L\beta_1$, $L\beta_2$, $L\gamma_1$, and Ma_1 .

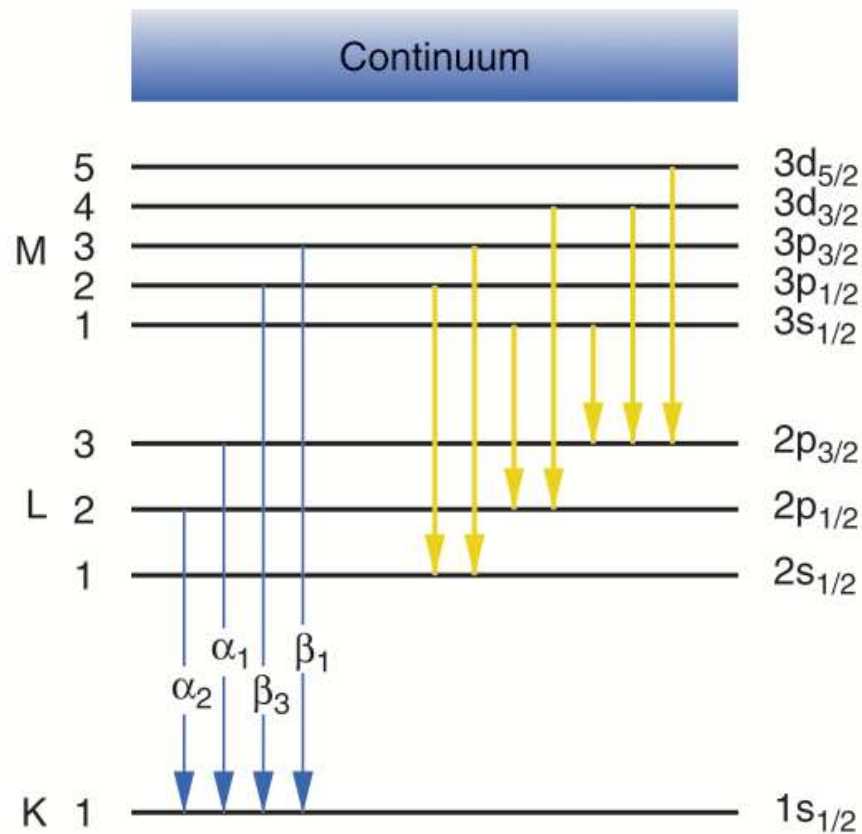


Figure 12. Transitions that make up the emission lines of X-ray fluorescence (Willmott, 2011).

A further advantage of this technique is that each point in an image carries a corresponding XRF spectrum, which provides pixel-by-pixel information on all measurable elements. However, a potential limitation arises from the type of substrate used for sample preparation, since substrates may fluoresce, saturating the detected signal or even masking the

presence of the same element within the aerosol particle. In general, synchrotron beamlines designed for nanoscale scanning X-ray Fluorescence Microscopy (nano-XRF) operate in the hard X-ray energy range (2–20 keV). This range is particularly advantageous because it eliminates the need for vacuum conditions during measurements. On the other hand, beam-induced damage is a common issue in this technique: while the spatial distribution of elements remains unchanged, the intense radiation can induce severe modifications in the chemical environment through photoionization of compounds. For this reason, when mutual spatial information is required in combination with other techniques, it is recommended to perform speciation measurements first to obtain more reliable results.

For source apportionment, bulk aerosol studies performed with benchtop instruments generally yield an average composition across all particles in the sample. Although such measurements are useful in many contexts, the analysis of individual particles and micro-aggregates requires nanometric resolution to accurately determine elemental concentrations within each aerosol. Nano-XRF therefore plays a crucial role in characterizing single particles and providing insights into their emission sources. In addition, the morphological information it provides is highly relevant, as particle size is a key factor in determining aging processes and atmospheric lifetimes (Heutte *et al.*, 2025).

2.2.5. X-ray Photoelectron Spectroscopy

The photon absorption phenomenon is also responsible for generating electron's emissions from the atoms, the analysis of their kinetic energies gives rise to photoemission spectroscopy. In 1914, the experiments conducted by Rutherford and his collaborators demonstrated that the kinetic energy of emitted photoelectrons depends on both the energy of the incident X-rays and the binding energy of the electrons (Hofmann, 2013). The detected radiation penetrates no more than a few atomic layers (approximately 0.5–10 nm), meaning that the emitted electrons originate from the near-surface region of the material. Therefore, this technique is particularly suitable for investigating the surface properties of solids (Ribeiro *et al.*, 2018).

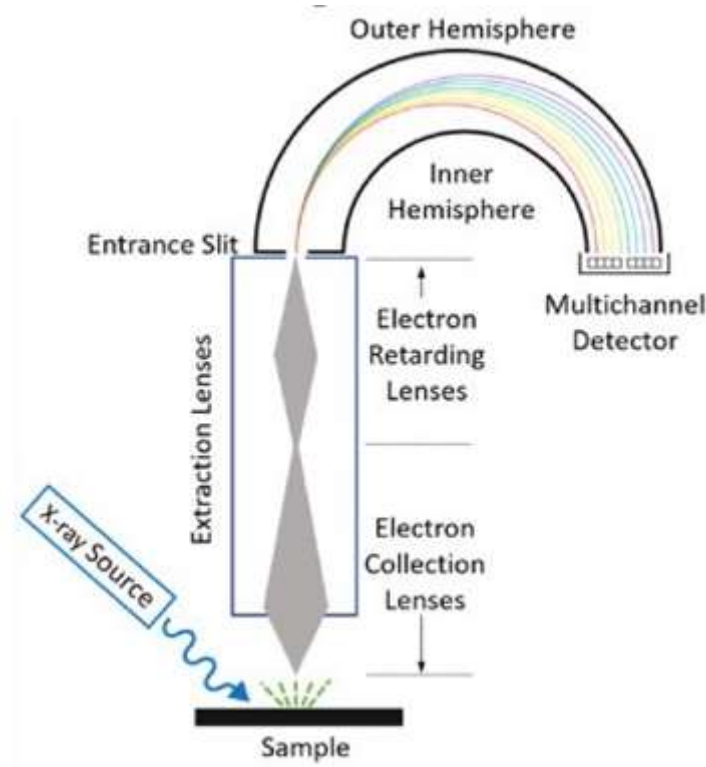


Figure 13. Schematic representation of an XPS experimental setup. An incident X-ray beam irradiates the sample, leading to the emission of photoelectrons, which are collected by a hemispherical analyzer to measure their kinetic energy and provide information about the surface chemical composition of the material (Stevie; Donley, 2020).

For the emission of a photoelectron to occur, the sample must be excited by a photon with energy $h\nu$. The photon energy is absorbed by the atom and transferred to one of its electrons. If this energy is sufficient to overcome the potential barrier that binds the electron to the atom, the electron is ejected. Although this process resembles X-ray absorption, what is actually measured in the XPS technique is the kinetic energy of the emitted electrons (Hüfner, 2003; Wagner *et al.*, 1979).

It is possible to determine the binding energy of the electrons (E_b) as the difference between the excitation X-ray photon energy (E_{photon}), the kinetic energy of the emitted electron (E_{kin}), and the work function of the analyzer (Φ_A). This relationship holds as long as the analyzer and the sample are at the same electrical potential, ensuring that the Fermi levels (E_F) of both are aligned. The mathematical expression can be described by (Hofmann, 2013):

$$E_b = E_{\text{photon}} - E_{\text{kin}} - \Phi_A \quad (6)$$

The photoelectron peaks are defined by the quantum number from which the electron originates, such that the orbital angular momentum L (s , p , d and f) and the spin angular momentum S result in a total angular momentum $J = L + S$. Each level with $J \geq 1$ is split into two sublevels, since $S = \pm \frac{1}{2}$. Accordingly, the intensity of these peaks can be determined by multiplying the corresponding degeneracy factor $2J + 1$ (Ribeiro *et al.*, 2018).

The XPS technique can be employed to determine elemental composition and quantities, material contamination, empirical formulas, binding energies of electronic states, the chemical (valence) state of elements in samples, and the thickness of different thin films (Hüfner, 2003; Wagner *et al.*, 1979). It is considered one of the most important experimental methods for investigating the electronic structure of molecules, solids, and surfaces (Ribeiro *et al.*, 2018).

The XPS spectrum is represented as a histogram of intensity versus binding energy. The elements present are identified directly from the positions of their characteristic binding energies, while information about surface composition is obtained by integrating the peak areas. Chemical speciation can also be performed through detailed analysis of the peaks, however, to extract this implicit information, the spectrum must be interpreted by means of peak fitting (Thompson *et al.*, 2001; Wagner *et al.*, 1979). To study the electronic structure of a sample, it is essential to evaluate the shifts in the photoelectron peaks and to have a thorough understanding of the material under analysis. A single chemical element can exist in multiple ionic or chemical states, and the positions of its photoelectron peaks may vary subtly depending on the surrounding chemical environment. Furthermore, in heterogeneous samples, peak overlap from different elements may occur, which complicates the analysis of the chemical species present (Ribeiro *et al.*, 2018).

2.2.6. Sample preparation of aerosols for synchrotron experiments

Each analytical technique requires specific sample preparation protocols, depending on the conditions necessary for detecting radiation–aerosol interactions. For instance, XPS analysis requires electrically conductive substrates to prevent surface charging effects that can distort the measured spectra. In contrast, infrared-based techniques rely on appropriate signal reflection or transmission conditions to ensure accurate detection of vibrational features (Fernando Amato-Lourenço *et al.*, 2024; Major *et al.*, 2020). Substrates commonly used in conventional aerosol characterization, such as PTFE, quartz, and glass, are not suitable for XPS

analysis. Their low electrical conductivity leads to surface charging effects, while their fibrous structure can result in high background noise and poor signal-to-noise ratios. To overcome these limitations, metallic substrates with smooth and conductive surfaces are preferred. Materials such as copper (Cu), silicon (Si), and aluminum (Al) may be used; however, they are prone to rapid oxidation or may interfere with the detection of the same elements present in the sample. Generally, gold-coated silicon (Si/Au) wafers are the most suitable substrates for such analyses.

In addition, transmission-based techniques present greater experimental challenges, as the combined thickness of the sample and substrate must typically be below ~ 400 nm. The most used substrates for such analyses are transmission electron microscopy (TEM) grids with a 400 mesh, typically made of Cu or Au, and coated with a carbon Type-B support film to retain aerosol particles across the grid openings.

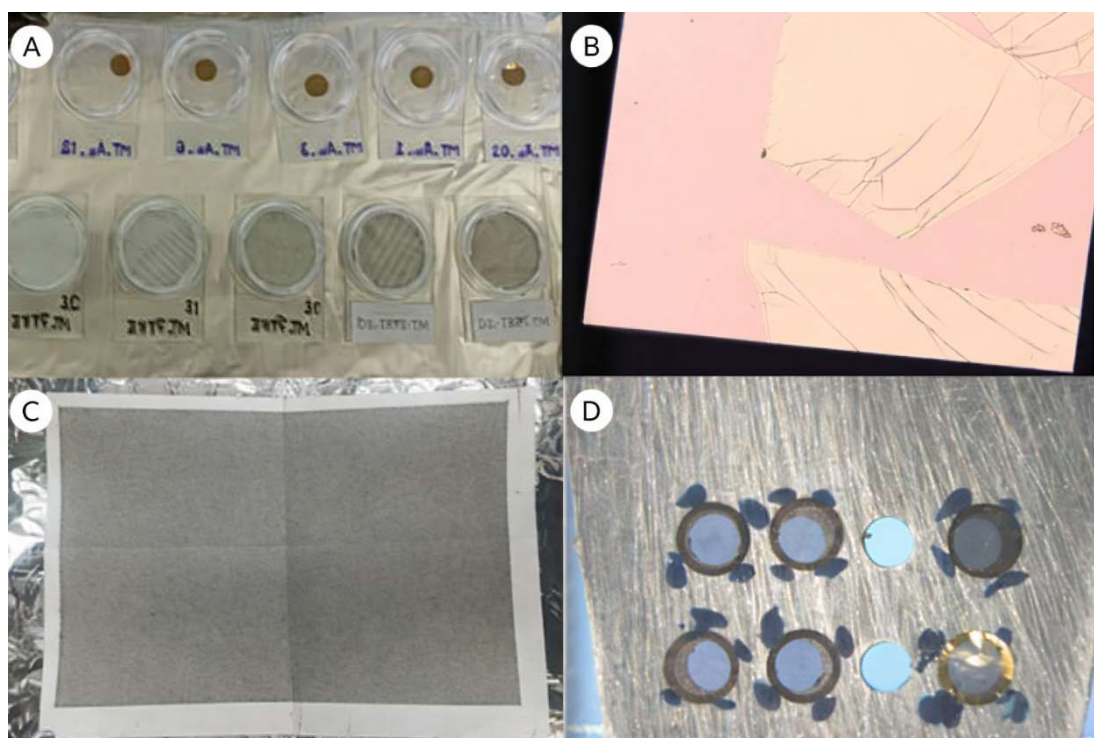


Figure 14. Sampling substrates and sample preparation materials used for aerosol characterization. (A) Si/Au substrates and Teflon filters employed for aerosol collection and synchrotron-based analyses. (B) Silicon nitride window used for STXM measurements. (C) Quartz filter containing a 24-hour aerosol sample collected in the Pantanal. (D) TEM grids (400 mesh), gold-coated with carbon Type-B support film, used for microscopy and spectromicroscopy analyses.

3. CHARACTERIZATION OF THE ELEMENTAL COMPOSITION OF AEROSOLS EMITTED IN THE DRY SEASON OF THE PANTANAL WETLAND, BRAZIL

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Article

Characterization of the Elemental Composition of Aerosols Emitted in the Dry Season of the Pantanal Wetland, Brazil

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Figure 15. Image featured on the cover of the issue in which the article was published.

ABSTRACT

The Brazilian Pantanal region experiences intense biomass burning during the dry season, releasing large quantities of gasses and particles into the atmosphere, which have serious implications on both the climate system and public health. Understanding the dynamics of these emissions is crucial for mitigating the impact on the ecosystem, its functioning, and potential anthropogenic disturbances. This study focused on analyzing emissions in the northern Pantanal during the 2022 drought. Concentrations of fine particulate matter (PM_{2.5}), black carbon (BC), and 25 chemical elements were measured using gravimetry, reflectance analysis, and X-Ray fluorescence, respectively, from samples collected between August and October 2022. The average concentrations of PM_{2.5} and BC increased approximately 4-fold and 2.5-fold, respectively, compared to averages from a decade ago. Significant increases were also observed in elements such as sulfur (S), potassium (K), iron (Fe), and silicon (Si). The maximum concentrations were comparable to values typical of the southern Amazon, a region known for high deforestation rates and land use changes. Elemental analysis revealed substantial shifts in concentrations, primarily associated with biomass burning (BB) and soil suspension. Additionally, enrichment factor (Ef) analysis showed that lead (Pb) levels,

correlated with human activities, were 200 times higher than those found under clean atmospheric conditions.

Keywords: AOD; EDXRF; biomass burning; black carbon; soil suspension

INTRODUCTION

Atmospheric aerosols are solid or liquid particles suspended in the atmosphere, ranging in size from approximately 0.001 to 100 μm . These particles play a critical role in the climate system, and their high spatial and temporal variability represents one of the greatest sources of uncertainty in Earth's radiative balance (IPCC, 2013; Kumar, Manish *et al.*, 2017). These uncertainties stem from a limited understanding of the physical and chemical properties of aerosols (Kang *et al.*, 2016; Kumar, K. Raghavendra *et al.*, 2014), which can affect the climate both directly by scattering and absorbing solar radiation (Rizzo, L. V. *et al.*, 2013; Thornhill *et al.*, 2018) and indirectly by modifying cloud properties, thereby influencing precipitation efficiency (Bennett *et al.*, 2019; Bond, T. C. *et al.*, 2013) and atmospheric reflectance. Certain aerosols, such as those containing carbon and mineral dust, have a high capacity to absorb radiation. Soot carbon particles, known as black carbon (BC), are emitted by biomass burning (BB), composed almost entirely of carbon, and significantly contribute to the absorption of solar radiation (Requia *et al.*, 2021), influencing atmospheric stability (Thornhill *et al.*, 2018). BC emissions result from various combustion processes, including biomass burning. The aging process of BC alters its lifespan and removal rate, complicating our understanding of its true role in the climate system (Bond, T. C. *et al.*, 2013). While aerosols generally have a cooling effect on the climate, counteracting the impact of greenhouse gasses, BC exerts a direct radiative forcing, making it one of the key contributors to global warming (IPCC, 2022).

In addition to the effects on the climate, emissions of particulate matter associated with climate change have directly impacted the health of the population in every region of the planet (Andrade Filho *et al.*, 2013). In Brazil, recent research has shown that biomass burning (BB) emissions significantly worsen hospital records related to respiratory diseases (Brunelli *et al.*, 2021). Local studies have also found a strong correlation between BB emissions and increased hospitalizations of children due to respiratory illnesses (Jacobson, Ludmilla da Silva Viana *et al.*, 2021), moreover, the association between meteorological conditions and aerosols increases

viral transmission and the risk of mortality from respiratory diseases (Arana, Andréa; Artaxo, 2014; Rizzo, L. V. *et al.*, 2011).

In Brazil, several studies have characterized the physical and chemical properties of aerosols in the central Amazon (Marengo *et al.*, 2021; Ponczek *et al.*, 2021b; Rizzo, Luciana Varanda *et al.*, 2018; Rizzo, L. V. *et al.*, 2013; Santos, Anna Carolinna Albino *et al.*, 2016; Saturno *et al.*, 2018). However, studies are still scarce in the south of the Amazon basin (Palácios *et al.*, 2022). This region, de-nominated as Pantanal Biome, is a unique ecosystem with peculiar characteristics due to flooding regimes, rich in biodiversity, and providing special conditions for monitoring the physical and chemical properties of aerosols. The Pantanal is an excellent laboratory for monitoring aerosols, as during the rainy season it can be representative of a practically clean atmosphere, under natural conditions. During the dry season, as in other areas of South America and Brazil, it is strongly influenced by emissions from biomass burning (Garcia *et al.*, 2021; Libonati *et al.*, 2022). The concentrations of atmospheric aerosols in the Pantanal northern region increase in the dry season. This is attached to anthropogenic activities such as biomass burning, gold mining, and carriage (Palácios *et al.*, 2022).

The Pantanal is the largest contiguous wetland in the world (Kumar, Sujay *et al.*, 2022; Martins *et al.*, 2022). Recent changes in land use and atmosphere conditions contributed greatly to the unprecedented forest fires recorded in the region, with approximately half of the area burned in 2020. This fire season was responsible for more than 3.9 million hectares burned with diverse impacts on the ecosystem, the hydrological cycle, and the economy (Guimarães *et al.*, 2018; Köppen; Geiger, 1936; Martins *et al.*, 2022). The occurrence of the 2020 burning motivated this study, which aims to carry out a specific chemical characterization of BB emissions in the Pantanal, now using the 2022 drought as a reference. The chemical results were also complemented with surface remote sensing analyses from Aerosol Robotic Network (AERONET) (Holben *et al.*, 1998).

MATERIAL AND METHODS

Study Site and Sampling

Aerosol samples were collected at the Base Avançada de Pesquisa do Pantanal (BAPP), located 160 km south of the capital Cuiabá–MT, in the SESC Pantanal Park–Baía das Pedras (16°29'56" S, 56°24'47" W), in the city of Poconé (Figure 16), and close to the SESC Private Natural Heritage Reserve (PNHR SESC). The Pantanal of Mato Grosso is located to the north

of the biome, the climate is classified as Humid Tropical with rainy summers and dry winters, *Aw* in the Köppen–Geiger classification (Guimarães *et al.*, 2018; Köppen; Geiger, 1936). Climatological factors make the Pantanal a unique ecosystem, such as the flat relief and seasonal rainfall dynamics, which contribute to flooding part of the biome. Considering only the cities of the Pantanal of Mato Grosso, the annual precipitation mean is approximately 1360 mm (Palácios *et al.*, 2022).

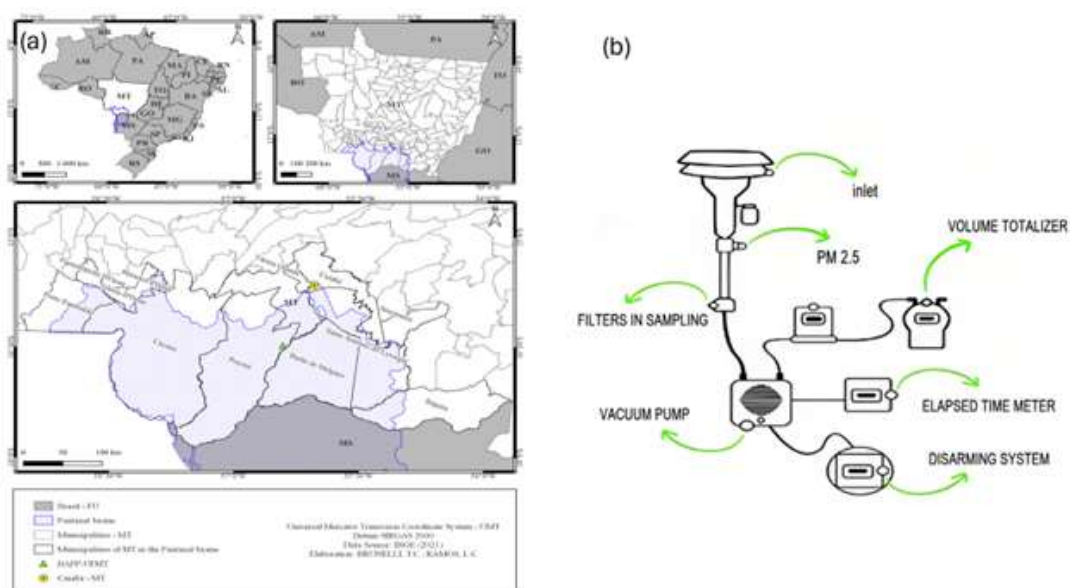


Figure 16. (a) Sampling site BAPP, Private Natural Heritage Reserve of SESC Pantanal–Baía das Pedras, state of Mato Grosso, north of Brazilian Pantanal, (b) Representation of the sampling system of atmospheric aerosol.

Particulate matter with an aerodynamic diameter smaller than $2.5 \mu\text{m}$ ($\text{PM}_{2.5}$) was collected using 47 mm Teflon membrane filters, which captured aerosols after the aerodynamic cut performed by the Partisol Model 2025i Sequential Air Sampler (Thermo Scientific, Waltham, MA, USA), operating at a flow rate of 17 Lmin^{-1} (Maenhaut *et al.*, 2005; McMurry, 2000). The sampling system is illustrated in Figure 16. Additionally, the system was automated, shutting down when the flow rate dropped below 16 Lmin^{-1} . Due to the challenging accessibility of the collection site, each filter was sampled for 7 days or until the system automatically shut down. Each sample was placed in a Petri dish, wrapped in aluminum foil, and stored in an inert environment to prevent damage or contamination of the samples. Similar interference conditions to those described in (Santos, Anna Carolinna Albino *et al.*, 2016), such as vehicular traffic on a nearby road, may have minimally influenced the sampling. Meanwhile, a complementary principal component analysis (PCA) (Veltkamp *et al.*, 1996) revealed that all

emissions could be explained by a single factor, reinforcing that the characterization in this study pertains to PM_{2.5} emissions primarily from biomass burning mixed with soil suspension. The collection campaign took place between 25 August 2022 and 14 October 2022 (2 months). Each filter represents a week average of PM_{2.5} we have separated into seven analysis periods: P1 = 08/25 to 09/01, P2 = 09/02 to 09/08, P3 = 09/09 to 09/15, P4 = 09/16 to 09/22, P5 = 09/23 to 09/30, P6 = 10/01 to 10/07, and P7 = 10/08 to 10/14. The period chosen for the sampling campaign, August to October, covers the critical period of emissions for biomass burning in the study area (Garcia *et al.*, 2021; Libonati *et al.*, 2022).

The samples were analyzed to determine the mass concentration of particulate matter in the atmosphere by gravimetric measurement. Before weighing, the samples had their static electricity removed by a U-shaped electrode (PRX U, Huang GmbH, Leinfelden Echterdingen, Germany). The procedure consists of determining the filter mass before and after sampling using an electronic microanalytical balance of nominal precision 1 µg (XP6U, Mettler Toledo, Greifensee, Switzerland) in a controlled environment with a temperature of 20°C and relative humidity of 50% (Arana, Andréa; Artaxo, 2014). The gravimetric method has an estimated precision of 90%, and the detection limit for the aerosol mass concentration is 0.3 µg m⁻³ (Artaxo *et al.*, 2002). The determination of BC concentration was carried out by optical reflectance analysis. The smoke stain reflectometer (M43D, Evans Electroselenium Ltd., Harlow, United Kingdom), which uses diffuse white light and a detector with a peak efficiency of around 550 nm, was calibrated with standard BC samples. The data collected from the reflectometer measures is applied to Equation (1) to determine the equivalent concentration of black carbon (BC) (Morais, Fernando Gonçalves, 2022).

$$BC = [(88.3 - (77.5 * \log R)) + (16.7 * (\log R)^2)] * A/V \quad (1)$$

Where R is the measured reflection, A is the filter area (in this case, 13.85 cm²), V is the volume of air in m³, and BC concentration is given in µg m⁻³.

Elemental analysis was performed through Energy Dispersive X-ray Fluorescence (EDXRF) with a spectrometer (EDX-700HS, Shimadzu, Kyoto, Japan). The sample was irradiated from below with X-rays. The fluorescence radiation is proportional to the quantity of each element, so detecting each element's energy condition allows for qualitative and quantitative analyses. The calibration of the fluorescence equipment (Epsilon 5, PANalytical, Almelo, Netherlands) can be found at (Arana, Andréa Araújo, 2014). Teflon filters were submitted to EDXRF, and spectra were accumulated for 900 s under the following conditions:

Al filter, vacuum as X-ray path, 10 mm diameter collimator, 10–20 keV energy range, 50 kV tube voltage, an Rh X-ray tube, and a Si (Li) detector (Vieira *et al.*, 2023). The analysis determined the concentration of 25 chemical elements (Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Cd, Sb and Pb) in the samples. The elementary analysis made it possible to calculate the mass concentration of the Dust, according to Equation (2), where each element concentration is given in $\mu\text{g m}^{-3}$ (Maenhaut *et al.*, 2005).

$$\text{Dust} = 1.16 * (1.90\text{Al} + 2.15\text{Si} + 1.41\text{Ca} + 1.67\text{Ti} + 2.09\text{Fe}) \quad (2)$$

Complementary Measures and Methods

To complement the discussions, this work used records of fire outbreaks (FO) and the sum of fire radiative power (FRP) from the National Institute for Space Research, INPE over the Pantanal (available at: <https://queimadas.dgi.inpe.br/queimadas/bdqueimadas> (accessed on 1 September 2024)). The measurements of air temperature (Ta), relative humidity (RH), accumulated rain (AR), net radiation (Rn), and wind direction and intensity were obtained from the BAPP meteorological tower observatory. Measurements from the CUIABA-MIRANDA station of the AERONET network (Holben *et al.*, 1998) were also used to obtain the optical depth for the fine, coarse, and total fractions of the aerosol (version 3: SDA Retrieval Level 2.0 data) (Sena; Artaxo; Correia, 2013). The Spearman correlation was used to evaluate the relationships between PM_{2.5} and BC concentrations with meteorological parameters and aerosol optical depth (AOD) values with a significance level of 95% (p-value < 0.05).

To quantify the anthropogenic influence on elemental concentrations, we adopted the enrichment factor (Ef) method, which quantifies the contribution of each element to a crustal condition (Duce; Hoffman; Zoller, 1975; Santanna *et al.*, 2016; Zoller; Duce, 1974) in the case of our study for background conditions. The enrichment factor (Ef) was calculated with Equation (3).

$$\text{Ef} = (\text{X/Al})_{\text{aerosol}} / (\text{X/Al})_{\text{background}} \quad (3)$$

Where (X/Al) aerosol and (X/Al) background refer to the ratio between the concentration of element X for Al in the atmosphere and element X for Al in the average background mass, respectively. Al is typically used as a reference because it represents more than 8 percent of the average crustal material (Duce; Hoffman; Zoller, 1975; Santanna *et al.*, 2016; Zoller; Duce, 1974).

The average elemental composition for the central Amazon, as reported by Arana and Artaxo (Arana, Andréa; Artaxo, 2014), was used as the background elemental composition. Elements with E_f close to 1.0 have a strong natural component while elements with high enrichment factors tend to be of artificial origin (Braga, C. F. *et al.*, 2005; Marcazzan *et al.*, 2001). These reference concentrations are justified by the unique conditions of elemental concentration in the central Amazon during the rainy season. Such conditions are closer to an atmosphere as clean as in non-anthropized areas (Arana, Andréa; Artaxo, 2014). Therefore, the E_f method highlights the anthropogenic influence on the composition of atmospheric aerosols and provides a valuable parameter for gaining insights into the sources of emissions.

RESULTS AND DISCUSSION

Variations in PM_{2.5} and BC Concentrations

The variations in PM_{2.5} concentrations for the respective analysis periods are presented in Figure 17 and detailed in Table 1. PM_{2.5} concentrations ranged from approximately $7 \mu\text{g m}^{-3}$ in period P4 to more than $80 \mu\text{g m}^{-3}$ in period P5, with an overall average of $36.62 \pm 31.69 \mu\text{g m}^{-3}$. The variation value represents the standard deviation from the mean, and each individual error in PM concentration is estimated at 10%, as cited in the methods. The variations in the concentrations of black carbon (BC) and dust were also evident in Figure 17, exhibiting a similar pattern to that of PM_{2.5}. These results can be explained by the meteorological patterns shown in Figure 18. Although the total analysis period is classified as dry, precipitation was recorded in periods P4, P6, and P7. Figure 18A illustrates the relationship between accumulated precipitation and aerosol load in the atmospheric column, revealing a distinct pattern of aerosol optical depth (AOD) compared to PM_{2.5} measurements at the surface. The total AOD measured at 500 nm is nearly equal to the values for fine particulate matter at 500 nm (AOD_{fine}), which is characteristic of biomass burning emissions. In this case, we observe an increase in AOD from periods P1 to P4, followed by a significant reduction that reaches a minimum AOD in P6, with values close to 0.4, which are typically found during the rainy season in this region (Palácios *et al.*, 2022).

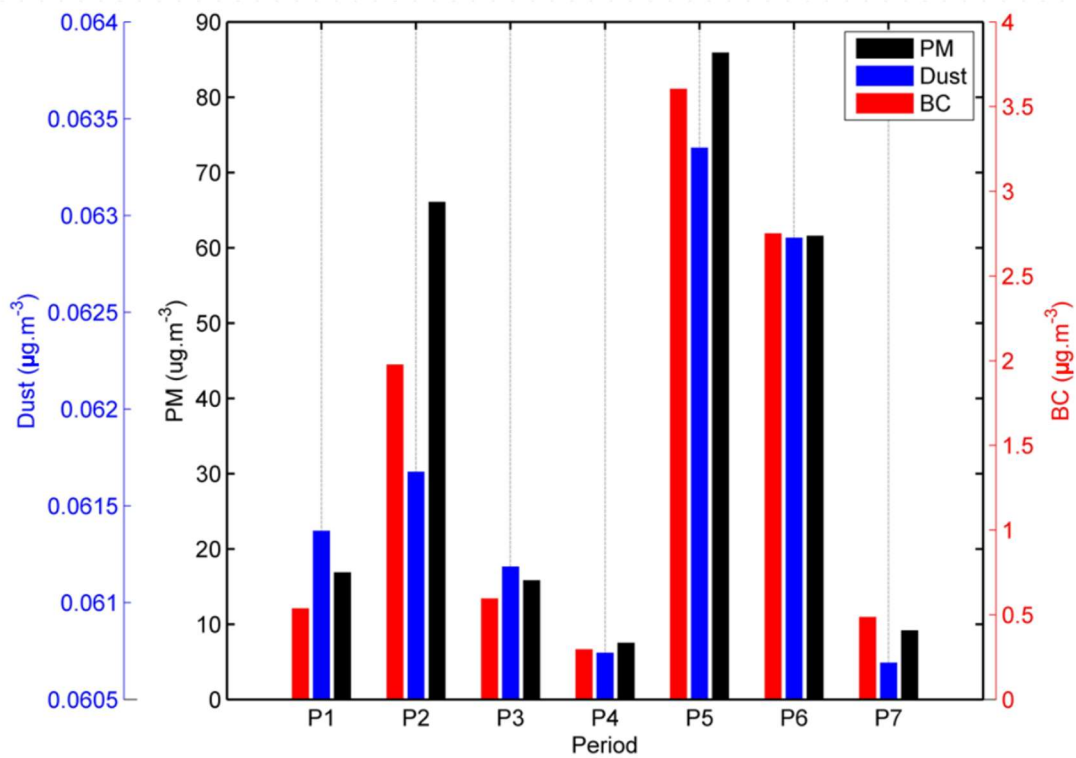


Figure 17. Concentration variation of $\text{PM}_{2.5}$, BC, and Dust concentration ($\mu\text{g m}^{-3}$) measured in the BAPP, between August and October 2022.

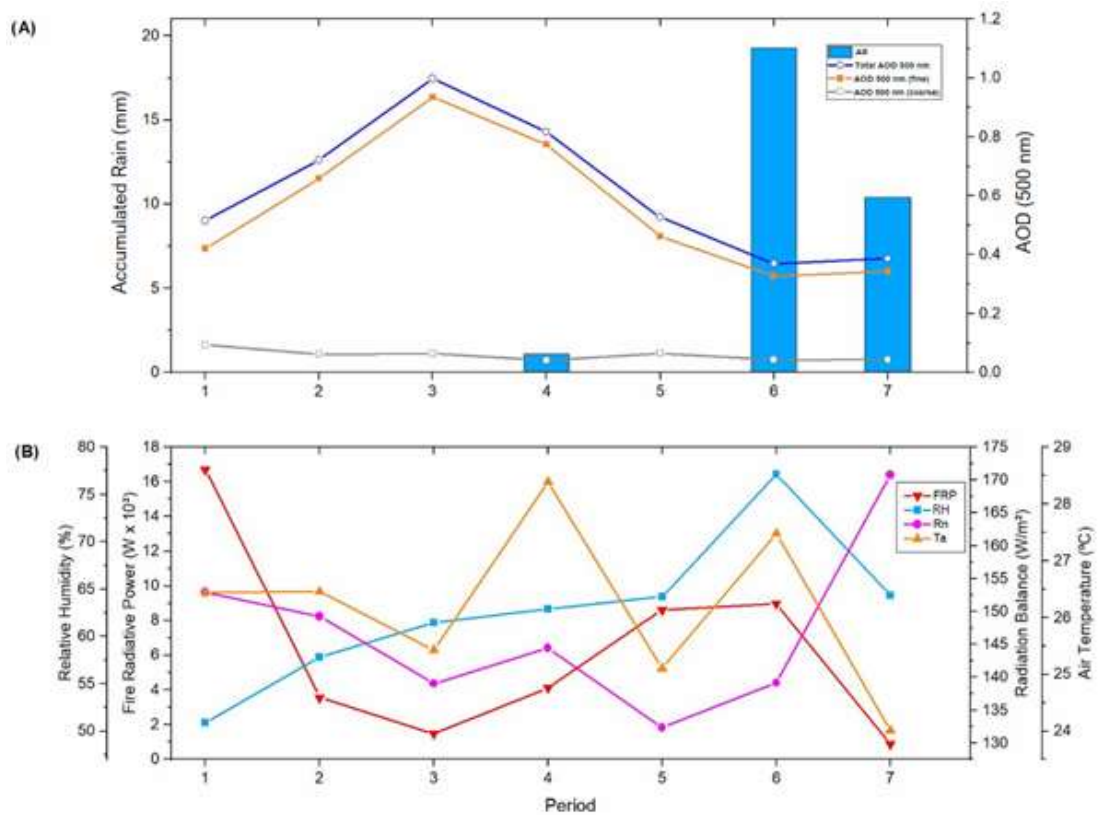


Figure 18. (A) Variation of Aerosol Optical Depth (AOD) 500 nm, Accumulated Rain (AR), and (B) meteorological conditions for the analysis period, where FRP, RH, Rn, and Ta stand for Fire Radiative Power, Relative Humidity, Net Radiation, and Air Temperature.

Table 1. Mean concentrations, standard deviations, minimums, and maximums associated with PM_{2.5}, BC, and elemental concentration for the entire study period (PM_{2.5} and BC are expressed as $\mu\text{g m}^{-3}$ and elements expressed as ng m^{-3}).

	Fine Particulate Matter BAPP Pantanal (Dry Season)			
	Mean	Σ	Min	Max
PM	36.62	31.69	7.02	83.66
BC	1.83	1.65	0.37	3.72
Na	94.05	95.33	19.68	279.87
Mg	28.12	27.45	0.86	81.81
Al	167.60	113.47	53.82	377.72
Si	243.52	181.99	71.59	500.15
P	33.54	38.88	5.74	98.82
S	688.32	627.43	200.90	1693.09
Cl	2.19	3.13	0.05	8.55
K	582.71	524.06	106.69	1392.17
Ca	49.07	45.44	11.56	132.15
Ti	18.93	21.01	0.02	53.30
Cr	2.05	1.71	0.42	4.62
Mn	3.86	3.30	0.74	8.65
Fe	238.28	172.63	56.47	582.40
Ni	0.53	0.49	0.11	1.45
Cu	2.65	3.15	0.69	9.16
Zn	7.66	7.22	1.33	22.49
As	0.14	0.10	0.01	0.31
Se	0.12	0.16	0.00	0.45
Br	8.62	7.47	2.36	18.84
Rb	0.98	0.80	0.20	2.53
Sr	2.27	3.96	0.00	11.00
Cd	8.56	8.48	0.85	22.37
Sb	5.47	4.55	1.78	13.39
Pb	4.28	9.16	0.08	24.93

The variation in surface concentrations has different dynamics than the aerosol load in the atmospheric column because wet deposition can act differently at each specific scale. Nevertheless, the precipitation is not enough to extinguish the burning spots, which continue to emit gases and particles that remain concentrated primarily in atmospheric regions closer to the surface. Therefore, the concentration of PM_{2.5} in the dry period will directly depend on the local burning and the influences of wind intensity and direction.

At a comparative level, the work of Santos et al. (Santos, Anna Carolinna Albino *et al.*, 2016), carried out on the same site and characterization techniques, found maximum PM_{2.5} concentrations ranging from 14 to 20 $\mu\text{g m}^{-3}$ during the dry period of 2012. Our results show an increase of approximately four times for the maximum concentrations of fine aerosols in the Pantanal. The work of Artaxo et al. (Artaxo *et al.*, 2013), south of the Amazon biome in a region directly affected by BB emissions, found maximums varying between 300 and 350 $\mu\text{g m}^{-3}$ in

the 2010 drought. The study by Santanna *et al.* (Santanna *et al.*, 2016), with measurements close to the urban area of Cuiabá, found maximum values that varied between 50 and 60 $\mu\text{g m}^{-3}$ in August 2004.

On average, $\text{PM}_{2.5}$ concentrations for the central Amazon during the dry period vary between 3.4 (Artaxo *et al.*, 2013) and 4.8 $\mu\text{g m}^{-3}$ (Arana, Andréa; Artaxo, 2014). For the south of the Amazon, in the arc of deforestation, this average goes to 33 $\mu\text{g m}^{-3}$ (Artaxo *et al.*, 2013). The average found in our study approximates the $\text{PM}_{2.5}$ concentrations of regions highly impacted by changes in land cover due to deforestation (Artaxo *et al.*, 2013). It is important to note that the averages obtained in our study is based on a relatively small sample size, and these average values could shift with a higher frequency of sample collection. Nonetheless, the analyses reveal elevated concentrations of $\text{PM}_{2.5}$ during the dry period of 2022.

For BC concentrations, our results also show higher concentrations than Santos *et al.* (Santos, Anna Carolinna Albino *et al.*, 2016) in the Pantanal. For the 2012 dry period, BC maximums varied between 1.6 and 1.8 $\mu\text{g m}^{-3}$, while our results show maximums of 3.7 and 2.8 $\mu\text{g m}^{-3}$ in periods P5 and P6, respectively. Recent work by Palácios *et al.* (Palácios *et al.*, 2022) also over the north of the Pantanal, with aethalometer measurements, found daily values above 3 $\mu\text{g m}^{-3}$ for the dry periods of 2017 and 2019, years in which the Pantanal was also heavily impacted by local fires (Garcia *et al.*, 2021). The mean for BC in our results was $1.83 \pm 1.65 \mu\text{g m}^{-3}$ while Palácios *et al.* (Palácios *et al.*, 2022) obtained 1.01 ± 0.95 , and $0.90 \pm 0.81 \mu\text{g m}^{-3}$, for the years 2017 and 2019, respectively. These differences can be justified by the differences in the methods of obtaining BC. Table 2 shows a comparison of our results for $\text{PM}_{2.5}$ and BC with other averages obtained for the Amazon in urban areas, both containing aerosol carbon sources such as biomass and fossil fuel burning.

Table 2. Comparison of means and standard deviations of $\text{PM}_{2.5}$ and BC ($\mu\text{g m}^{-3}$) for different regions in Brazil.

PM_{2.5}	BC	Local	Period	Reference
3.40 ± 2.00	0.23 ± 0.15	ZF2 Amazon Forest	2008–2012	Artaxo <i>et al.</i> , 2013)
33.00 ± 36.00	2.80 ± 2.92	PVH Amazon deforested	2009–2012	(Artaxo <i>et al.</i> , 2013)
1.65 ± 0.92	0.09 ± 0.06	ZF2 Amazon Forest	2008	(Arana, Andréa; Artaxo, 2014)
7.60 ± 3.70	1.20 ± 0.80	Cuiabá	2004	(Santanna <i>et al.</i> , 2016)
26.72 ± 14.20	2.27 ± 1.30	São Paulo	2019	(Vieira <i>et al.</i> , 2023)
8.66 ± 3.14	0.76 ± 0.42	Pantanal	2012	(Santos, Anna Carolinna Albino <i>et al.</i> , 2016)
--	0.75 ± 0.83	Pantanal	2017–2019	(Palácios <i>et al.</i> , 2022)
36.62 ± 31.69	1.83 ± 1.65	Pantanal	2022	This work

All information in Table 2 was extracted for the fine fraction of aerosols in the period considered dry, that is, with the influences of BB emissions. The results show large variations between $PM_{2.5}$ and BC values which can be explained by several factors, such as the acquiring method, characteristic local emission, and the sampling period. Regarding the sampling period, we highlight that for El Niño years, the central and northern regions of Brazil are influenced by a precipitation deficit, causing an increase in the dry period resulting in larger burned areas and more emissions of aerosols. The study by Palácios et al. (Palácios *et al.*, 2024) showed that for El Niño conditions, there is a significant increase in the aerosol load south of the Amazon basin. The increase in BC concentrations found in this study may have a direct influence on the local microclimate, giving positive feedback on temperature maximums (IPCC, 2022). The study by Curado et al. (Curado *et al.*, 2024) in the Pantanal showed a positive correlation between BC and temperature maximums with consequences for carbon capture.

Meteorological Influences

Figure 18 shows the average meteorological conditions for each period of analysis. Although there is no direct relationship, it is possible to observe some relationships between AOD_{total} , $PM_{2.5}$, and BC concentrations. AOD_{total} values were above 0.4 in the initial five periods, and the third period presented the highest AOD_{fine} average, 0.93. We emphasize that the AOD_{fine} is linked to the amount of fine optically active aerosols in the atmosphere, therefore a good correlation with $PM_{2.5}$ is expected. However, differences in the observed patterns emerged across the analyzed periods. Between P1 and P2, AOD_{fine} and $PM_{2.5}$ exhibited similar behaviors. In the P3 onward, surface concentrations decreased while AOD_{fine} continued to increase. The patterns of air temperature and relative humidity mirrored the surface measurements (Palácios *et al.*, 2024). Air temperature gradually increased from P1 to P2 before declining in P3. These findings are consistent with the positive correlation between BC concentration and maximum air temperature.

Figure 18B also shows the average behavior of the R_n . The behavior of R_n has no relationship with AOD_{total} or surface concentrations because this interaction is complex (Huang *et al.*, 2021b; Palácios *et al.*, 2024). Blocking direct solar radiation, through scattering or absorption, can negatively influence R_n , however, retention of this radiation in the atmosphere can increase the amount of long-wave radiation, influencing R_n positively (Curado *et al.*, 2024; Palancar *et al.*, 2016). In general, more measurements in a specific experimental design need to be developed in the Pantanal so that these feedback processes are better understood. In this study, we justify the main variations in surface concentrations based on the number of burning

spots that occurred during the analysis period. Fire Outbreaks (FO) is a recurring problem in the Pantanal due to the environmental impact and the damage to health they can cause (Caumo; Lázaro; *et al.*, 2022; Libonati *et al.*, 2022). During the dry season, fires are constant as shown in Figure 19, and with the contribution of wind intensity and direction (Figure 20), concentrations can still increase due to transport in the atmosphere (Palácios *et al.*, 2022).

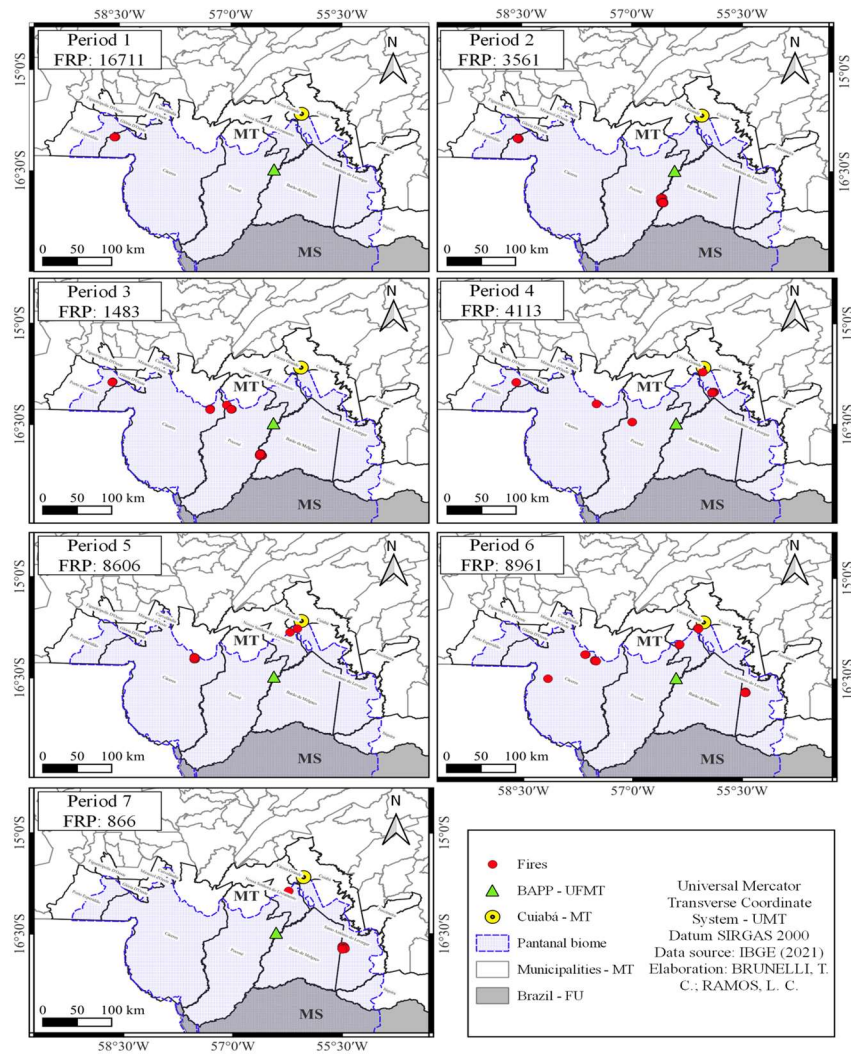


Figure 19. Geographical distribution of fires by period in the municipalities of Mato Grosso in the Pantanal biome between August and October 2022.

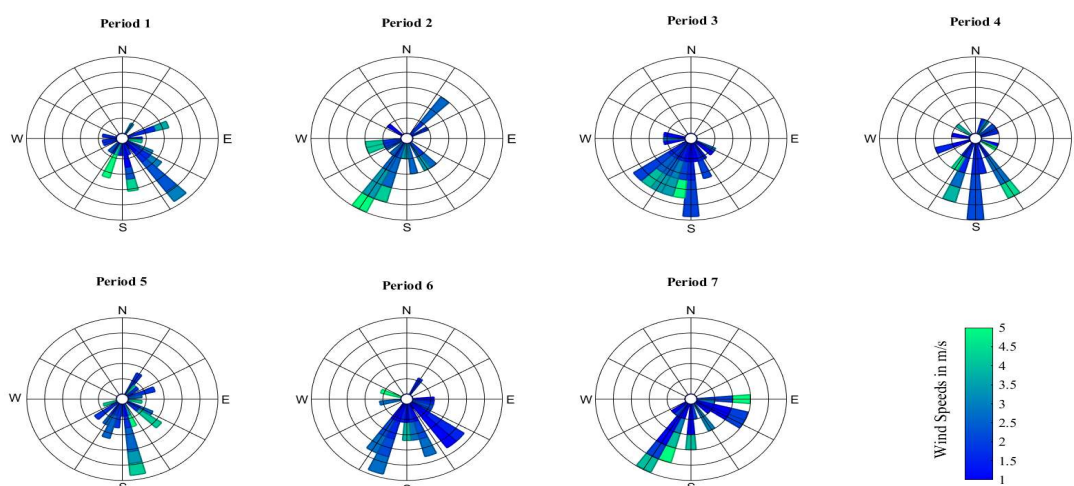


Figure 20. Roses of wind indicating the mean velocity and direction of the wind throughout the period between August and October 2022 measured at the micrometeorological tower in the BAPP.

Our analysis shows that P1 obtained the highest value for FRP, reaching 16,711 W, however, the distance from BAPP, FO (Figure 19), wind direction (Figure 20), and $PM_{2.5}$ concentration (Figure 17) suggest low BB influence on collected samples. The largest $PM_{2.5}$ peaks occur in the second, fifth, and sixth periods, with the values of FRP 3561, 8606, and 8961 W, respectively. Considering the position of the FO and the wind speed and direction, the samples collected in these periods may have been directly influenced by regional forest fires. Furthermore, period five (P5) presented the highest number of FO and at the same time the maximum dust concentration of $3.7 \mu g m^{-3}$ (Figure 17). When analyzing the correlations between surface concentrations ($PM_{2.5}$ and BC) with the other variables used in this study, no statistically significant correlation was found. The similarity in the variation of black carbon (BC) and dust concentrations suggests that aerosol composition in the northern Pantanal during the dry season is a mixture of biomass burning and soil resuspension. The PCA analysis confirmed that 99% of the emissions could be explained by a single factor.

In addition, the wind intensity contributes to soil suspension. Crossed with other factors such as land use and models of particle dispersion, it determines some aspects of aerosol composition and their trajectory (Garba; Abubakar, 2018), however, this work is focused on the characterization of $PM_{2.5}$, thus a qualitative analysis of air conditions and elemental concentration was enough. Despite this, the spatial and temporal variability of aerosols over a long 1 week must influence the correlation between $PM_{2.5}$ concentrations and the other meteorological variables. Precipitation events should decrease the concentration of PM in the atmosphere (Possanzini; Buttini; Di Palo, 1988), such as the variation observed in P6 to P7.

Nonetheless, the continuous emissions of biomass and soil suspension could rapidly increase the concentrations of PM_{2.5}, BC, and Dust.

Elemental Concentration and Enrichment Factor

Figure 21 presents the most significant elemental concentrations observed during the study period. In comparison with the study by Santos et al. (Santos, Anna Carolinna Albino *et al.*, 2016), an increase in the concentrations of sulfur (S), potassium (K), iron (Fe), and silicon (Si) is noticeable. While the concentrations of Fe and Si are close to those measured in 2012, the maximum concentrations of S and K were three times higher. Elemental concentrations follow the same pattern as PM_{2.5} and BC concentrations with small variations for Si and Fe. For Si, concentrations were the same in periods P5 and P6, while Fe suffered an approximate reduction of 50% for the respective periods. The study by Santanna et al. (Santanna *et al.*, 2016) explains that the increased concentrations of aluminum (Al), silicon (Si), and iron (Fe) during the dry season are primarily linked to soil conditions, as these elements are characteristic of crustal material and among the most abundant in Earth's crust. During this period, arid soil, combined with higher average wind speeds and minimal precipitation, transforms the soil into a significant emission source, contributing to aerosol levels alongside biomass burning.

Although the factor analysis was limited by the sample quantity, the dominant elemental concentrations still show a strong association with biomass burning emissions, with additional contributions from soil resuspension (Arana, Andréa; Artaxo, 2014; Artaxo *et al.*, 2013; Santanna *et al.*, 2016; Santos, Anna Carolinna Albino *et al.*, 2016). In fact, 99% of the emissions were attributed to a single factor that we identify as a mixture of BC and Dust. The concentrations of Fe, S, and K can be a good indication of BB contribution. The classic study by Andreae (Andreae, M.A., 1983) highlights that the concentrations of K can be used as an indicator of fires in the flaming phase. However, Urban et al. (Urban *et al.*, 2012) highlight the limitations of using K as a marker when studying soil resuspension emissions in fertilized areas.

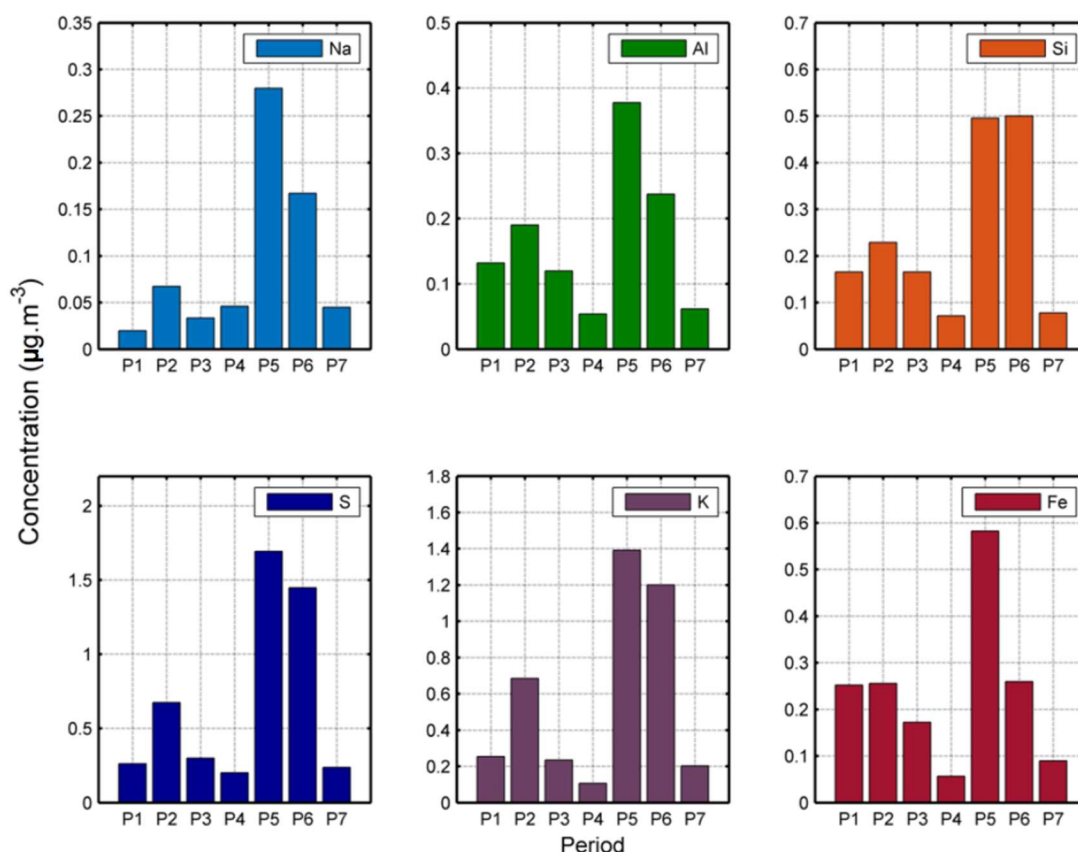


Figure 21. Variation in the mean concentration of major chemical elements during the study period.

For the heavy metals Pb and Cd, the averages found in our study were 4.28 and 8.56 ng m⁻³. In the case of Pb, the maximum concentration reached 24.93 ng m⁻³. This peak value occurred in P5 and is six times higher than the average concentration for this element (see Table 1). The presence of Pb may be related to anthropogenic factors, especially mining activities that contaminate the soil with heavy metals and increase the suspension of contaminated soil (Urban *et al.*, 2012). The dust compound, calculated from the concentration of the elements Al, Si, Ca, Ti, and Fe (Equation (2)), is the main constituent of natural atmospheric aerosols. Annually, the Amazon receives a substantial load of dust from the African continent, which transports tons across the Pacific Ocean (Garba; Abubakar, 2018). In the Pantanal, land use by agricultural activities, mining, and vehicle traffic is responsible for the suspension of dust, which, depending on the composition of the soil, can pose health risks (Caumo; Yera; *et al.*, 2022; Santos, Anna Carolinna Albino *et al.*, 2016). This work calculated the maximum dust concentration at 3.7 µg m⁻³, while in the study by Morais (Morais, Fernando Gonçalves, 2022) during the dry season at the Amazon Tall Tower Observatory (ATTO), the maximum concentration was 1.4 µg m⁻³, highlighting that areas with greater concentration of human activities promote greater soil suspension.

Reference measurements from Arana and Artaxo (Arana, Andréa; Artaxo, 2014) facilitated the determination of the enrichment factor (Ef) for 17 elements, as illustrated in Figure 22. The results in Figure 22 show that for all evaluated elements, there is an anthropogenic contribution. For the fine fraction of aerosols, BB emissions are responsible for the high values of K, S, Mg, and Zn (Artaxo *et al.*, 2002; Reid *et al.*, 1998). In this case, we highlight the Ef of K which was approximately 10 times higher than the natural concentration. As for soil resuspension emissions, we have increases in Al, Si, Ti, Fe, Ni, and Cu, which varied in elevations of 1.6 for Si and 12.8 for Cu (times higher). The results also show that Pb was the most anthropic contribution element in the study with Ef above 200 times the natural emission value, although its average concentration is low (see Table 1). The results identified in this study are generally consistent with those obtained by Santanna *et al.* (Santanna *et al.*, 2016), although the values are approximately 10 times higher for most elements. The concentrations of Cl, K, and Br can be attributed to biomass burning, while Fe, Cu, Zn, and Pb suggest anthropogenic emissions. This indicates that the region is undergoing significant anthropogenic transformation, which may have serious implications for the environment and public health. As previously mentioned, these transformations can be directly associated with mining activities that contaminate the soil with heavy metals and increase the suspension of contaminated soil (Garba; Abubakar, 2018).

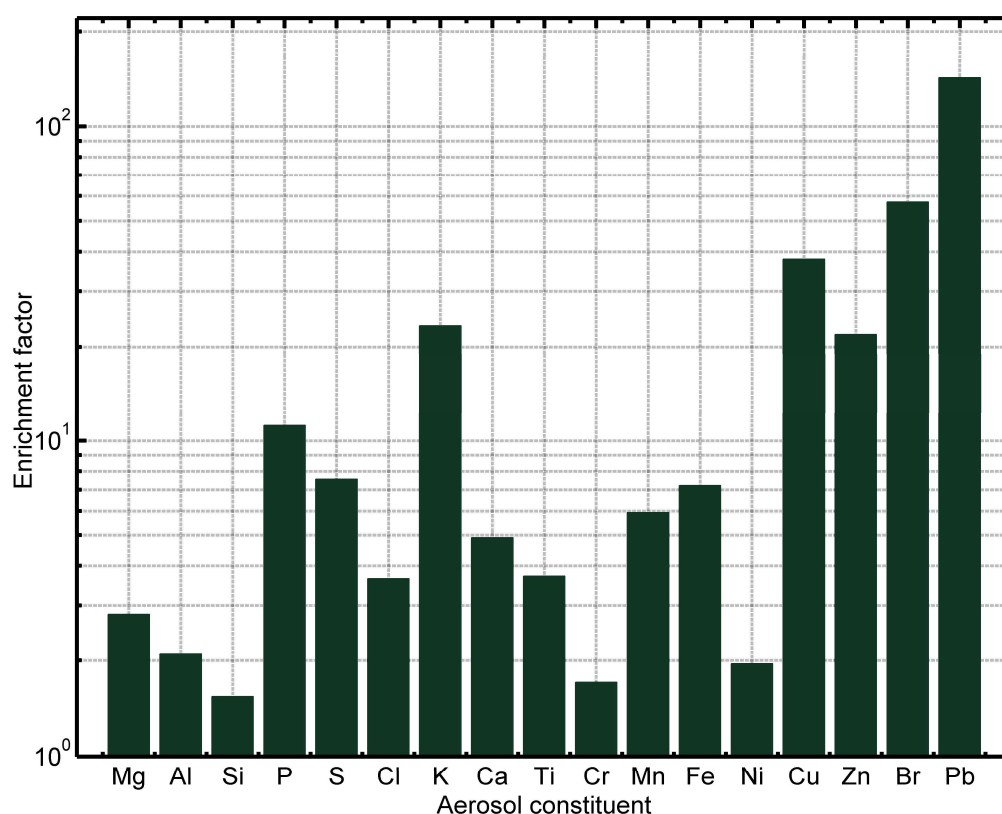


Figure 22. Enrichment factors (Ef) for the PM_{2.5} fraction. Enrichment factors close to 1 indicate a natural source while enrichment factors > 1 indicate an anthropogenic source.

CONCLUSIONS

In this study, the mass concentrations of PM_{2.5} and BC were determined through gravimetric analysis and optical reflectance during the dry period of 2022 in the northern Pantanal. Concentrations varied across the analyzed periods, reflecting local BB emissions. The average concentration of PM_{2.5} was $36.62 \pm 31.69 \mu\text{g m}^{-3}$, while BC had an average of $1.83 \pm 1.65 \mu\text{g m}^{-3}$. These concentrations represent increases of approximately 80% and 60% for PM_{2.5} and BC, respectively, compared to those recorded a decade ago at the same site. The elemental concentration of S, K, Fe, and Si has also increased significantly in the last decade. In this study, the concentration of those elements reached 688.32 ± 627.43 , 582.71 ± 524.06 , 238.28 ± 172.63 , and $243.52 \pm 181.99 \text{ ng m}^{-3}$, respectively. Additionally, other elements such as Pb, with a maximum concentration of 24.93 ng m^{-3} , and Cd, with a maximum concentration of 22.37 ng m^{-3} indicate a worrying concentration. Furthermore, a significant increase in heavy metals was found in the composition of aerosols. Notably, the average concentration of Pb, which was the most significant constituent in the enrichment analysis, exhibited values 200 greater than those

typically observed under clear atmospheric conditions. We also emphasized the enrichment factor values for Br and Cu. These compounds indicate a strong anthropogenic contribution, primarily emitted by biomass burning and gold mining. Environmental factors such as wind speed and low precipitation values contribute to soil suspension as an aerosol source, indicating that aerosols emitted in the dry season period are predominantly a mix of biomass burning and dust. The results, both in terms of elemental composition and BC concentration over the 10-year interval, indicate a shift in the regional dynamics of the Pantanal. We suspect that the increase in anthropogenic activities in the biome in the last decade is responsible for this change in the concentration and composition of atmospheric aerosols in the Pantanal Mato Grosso and we aim to explore this issue further in future research.

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Data Availability Statement: The AERONET website provides data analysis and dissemination tools at <https://aeronet.gsfc.nasa.gov> (accessed on 16 November 2022). Data can be viewed in the data display interface, acquired using the data download tool, analyzed, and downloaded using some analysis tools provided by AERONET. The INPE website provides data analysis and dissemination tools at <https://queimadas.dgi.inpe.br/queimadas/portal> (accessed on 20 November 2022). Every other data must be requested directly from the authors.

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4. MULTISCALE CHARACTERIZATION OF AMAZONIAN AEROSOLS REVEALS SIZE-DEPENDENT CHEMICAL GRADIENTS AND CLOUD ACTIVATION PROCESSES

This work highlights the powerful complementarity between advanced synchrotron-based techniques and conventional field measurements for atmospheric aerosol characterization. By providing access to physicochemical information at unprecedented levels of chemical specificity and spectral resolution, these techniques revealed features that cannot be fully captured through traditional approaches alone. The results demonstrate that significant opportunities remain for advancing our understanding of aerosol properties and atmospheric processes, emphasizing the potential of synchrotron science to open new research directions and contribute to future high-impact studies in atmospheric and environmental sciences.

ABSTRACT

This study presents a comprehensive multiscale characterization of size-segregated aerosols in the central Amazon during the wet season by integrating in situ measurements (SMPS, ACSM) with advanced laboratory techniques (FTIR, XPS, and EDXRF). The results reveal a pronounced size-dependent chemical gradient, where smaller particles are enriched in aromatic carbon and exhibit lower oxidation levels, while particles in the accumulation mode display enhanced surface oxidation and a higher abundance of oxygenated functional groups. This pattern is consistent with extensive atmospheric processing, likely including cloud-related transformations. Despite both sampling periods being dominated by highly oxygenated organic aerosol ($f_{44} \approx 0.22$), subtle compositional differences are observed. Period 1 exhibits relatively higher sulfate and equivalent black carbon contributions, suggesting a modestly stronger influence of long-range transported anthropogenic emissions, whereas Period 2 is more organic-dominated. These variations are consistent with the observed shift in the Hoppel minimum from ~ 90 nm to ~ 100 nm, indicating differences in cloud condensation nuclei (CCN) activation efficiency. Surface-sensitive XPS measurements further demonstrate that particle surface composition differs from bulk composition, highlighting the importance of mixing state and surface chemistry in controlling aerosol hygroscopicity and cloud activation. Overall, the findings demonstrate that even subtle variations in aerosol composition and mixing state significantly influence particle growth and CCN activation in the Amazon, emphasizing the

need for integrated surface- and bulk-resolved approaches to improve the representation of aerosol–cloud interactions in tropical environments.

INTRODUCTION

Atmospheric aerosols are significant influences of the global climate and directly affect the Earth's energy balance by absorbing and scattering incoming solar radiation. They also influence cloud properties indirectly, such as albedo and lifetime. The impact of these particles depends substantially on their chemical composition and concentration, which in turn influence their optical properties and hygroscopicity (Artaxo *et al.*, 2022; Seinfeld; Pandis, 2006; Zieger *et al.*, 2017). Additionally, aerosol size and morphology are key factors in predicting their behavior in the atmosphere, particularly regarding their efficiency as Cloud Condensation Nuclei (CCN) (Colbeck; Lazaridis, 2014; Pöhlker, Mira L. *et al.*, 2018; Santos, Luis F.E.D. *et al.*, 2022). Thus, the microphysics of atmospheric particles is essential for improving our understanding of the climate implications of both natural and anthropogenic emissions. Real-world samples are crucial due to the unique emission conditions and mixing states resulting from aerosol aging, which cannot be fully replicated in laboratory settings. Measurements of both real and laboratory-generated samples are complementary and necessary to capture the complexity of atmospheric aerosols (Daemne *et al.*, 2021; Santos, L. F.E.D. *et al.*, 2024; Virtanen *et al.*, 2025). Additionally, the particle size carries a lot of information regarding emission source and particle formation and aging in the atmosphere (Colbeck; Lazaridis, 2014). Investigating size-segregated aerosols is an interesting approach to evaluate and compare the composition, morphology, and environmental impact across different size ranges and their respective constituents. Moreover, combining conventional techniques with advanced aerosol characterization methods offers a unique opportunity to obtain complementary insights into these particles (Ault; Axson, 2017; Cai *et al.*, 2020; Kaushik *et al.*, 2021).

The role of the Amazon in climate regulation is widely recognized, yet it remains under continuous investigation due to its complexity (Artaxo *et al.*, 2022; Fernandes *et al.*, 2025), its influence on the hydrological and carbon cycles of South America is also well established (Malhi *et al.*, 2008). The cyclical characteristics of emissions during the dry and rainy seasons are pronounced; the varying contributions from emission sources throughout these periods

determine the composition and concentration of aerosols in the region (Arana, Andréa; Artaxo, 2014; Saturno *et al.*, 2018). The rainfall regime in South America is closely linked to emissions from the Amazon, and changes in this regime are already being observed as result of altered cloud formation. These changes are contributing to the increased duration and severity of the dry season, and we highlight the recent preoccupation with the drought and climate changes in the region (Da Silva *et al.*, 2019; Fernandes *et al.*, 2025). Climate projections indicate that even more significant shifts may occur in the coming years and an increase of extreme weather events (Da Silva *et al.*, 2019; Gra, 2004; IPCC, 2021; Ramanathan; Feng, 2008).

Recent studies in Amazon showed that upper troposphere could be an important source of tropospheric aerosols, in regions that are not strongly affected by anthropogenic or natural primary particles, also indicating that SO_4 and H_2SO_4 are not a strong source of new particles in the upper troposphere (Andreae, Meinrat O. *et al.*, 2018). Particles size-fraction of 60-80 nm are large enough to become CCN, however, smaller particles could also turn into CCN induced by sub-50nm particles ($\text{CN}_{<50}$) downdraft injection (Andreae, Meinrat O. *et al.*, 2018; Franco *et al.*, 2022). Other study of CCN over the Pacific suggest that larger nonvolatile aerosols associated with combustion and biomass burning suppressed and far exceeded natural volatile CCN (Clarke; Kapustin, 2010). Events of particle growth of $\text{CN}_{<50}$ aerosol particles mainly (88%) occurs between January and June (Franco *et al.*, 2022). During the wet season, Quiet New Particle Formation accounts for approximately 45% of sub-25 nm particles, highlighting its significant contribution to ultrafine particle populations (Meller *et al.*, 2025). Additionally, sub-40 nm particles also grow into the Aitken mode size range reacting with trace gases at mixed layer between 0 – 1200 m induced by O_3 and NO_2 but other mechanisms could produce CCN, mainly from anthropogenic sources (Machado *et al.*, 2024).

Our study contributes to the understanding of aerosol science in the Amazon by investigating particles processing through the analysis of size-segregated particles from a physicochemical perspective. Such information remains scarce in this region and provides valuable insights into the still enigmatic aspects of particle formation in the Central Amazon.

MATERIAL AND METHODS

Sampling Site

The sampling site was the Amazon Tall Tower Observatory (ATTO), located at 02.1483° S, 59.0033° W, approximately 150 km north of Manaus, the capital of the state of

Amazonia. This remote region of the Amazon rainforest has a canopy height of approximately 30 meters, annual precipitation exceeding 2300 mm, and an average yearly temperature of 27 °C, although climate change is causing an increase of about 0.1 °C per decade. The average altitude is around 130 meters above sea level, the terrain is typical of a lowland plain, and the climate is classified as Af – tropical rainforest climate according to the Köppen–Geiger classification (Franco *et al.*, 2022; Köppen; Geiger, 1936; Nobre; Sampaio; Salazar, 2007). This region experiences seasonal variations that can be divided into a dry season (June–November) and a wet season (December–May). During these periods, the aerosol population changes significantly due to shifts in emission source profiles and the presence of precipitation, which contributes to the removal of coarse particles through wet deposition (Artaxo *et al.*, 2002; Da Silva *et al.*, 2019; Franco *et al.*, 2022; Palácios *et al.*, 2022). Deforestation is a major issue in the region, with an estimated loss of approximately 68,000 km² of forest per year. However, during the sampling period, local emissions had limited influence, with aerosol composition being more strongly affected by long-range transport (Franco *et al.*, 2025; INPE, 2025; Malhi *et al.*, 2008).

During the wet season, aerosols are predominantly derived from biogenic emissions and are characterized by smaller particle diameters, primarily within the nucleation mode (< 30 nm) and accumulation mode (100–300 nm). These particles participate in key atmospheric processes such as coagulation and condensation with gaseous precursors, which contribute to their aging and growth in size (Mahilang; Deb; Pervez, 2021; Pöhlker, Christopher *et al.*, 2012; Pöschl *et al.*, 2010). Additionally, from January to August, a phenomenon known as the Intertropical Convergence Zone (ITCZ) contributes to the Amazonian aerosol population by transporting dust emitted from the Sahara Desert, biomass burning emissions from the African continent, and marine aerosols (Fernandes *et al.*, 2025; Holanda *et al.*, 2023, 2020; Parra *et al.*, 2024). Another key characteristic of aerosols during this period is their high potential to act as efficient cloud condensation nuclei (CCN), providing surfaces for water vapor condensation into droplets. These processes can enhance cloud formation and, under certain atmospheric conditions, contribute to precipitation in regions of South America (Artaxo *et al.*, 2022; Pöhlker, Mira L. *et al.*, 2018).

Sampling and *in-situ* measurements

This study collected aerosols by impaction method, a cascade impactor Micro-Orifice Uniform Deposit Impactor (MOUDI), model 122R Nano-MOUDI II Impactor (MSP Corporation, Shoreview, MN, USA) with 13 stages, starting air flow rate was adjusted to 30

LPM with an inlet located at 325 m above the ground in the top of ATTO. The sampling stages covered particle sizes ranging from the Aitken mode to the coarse mode, with aerodynamic cut-off diameters of 3.2–1.8, 1.8–1.0, 1.0–0.56, 0.56–0.32, 0.32–0.18, 0.18–0.10, 0.10–0.056, and <0.056 μm , as shown in Figure 23.

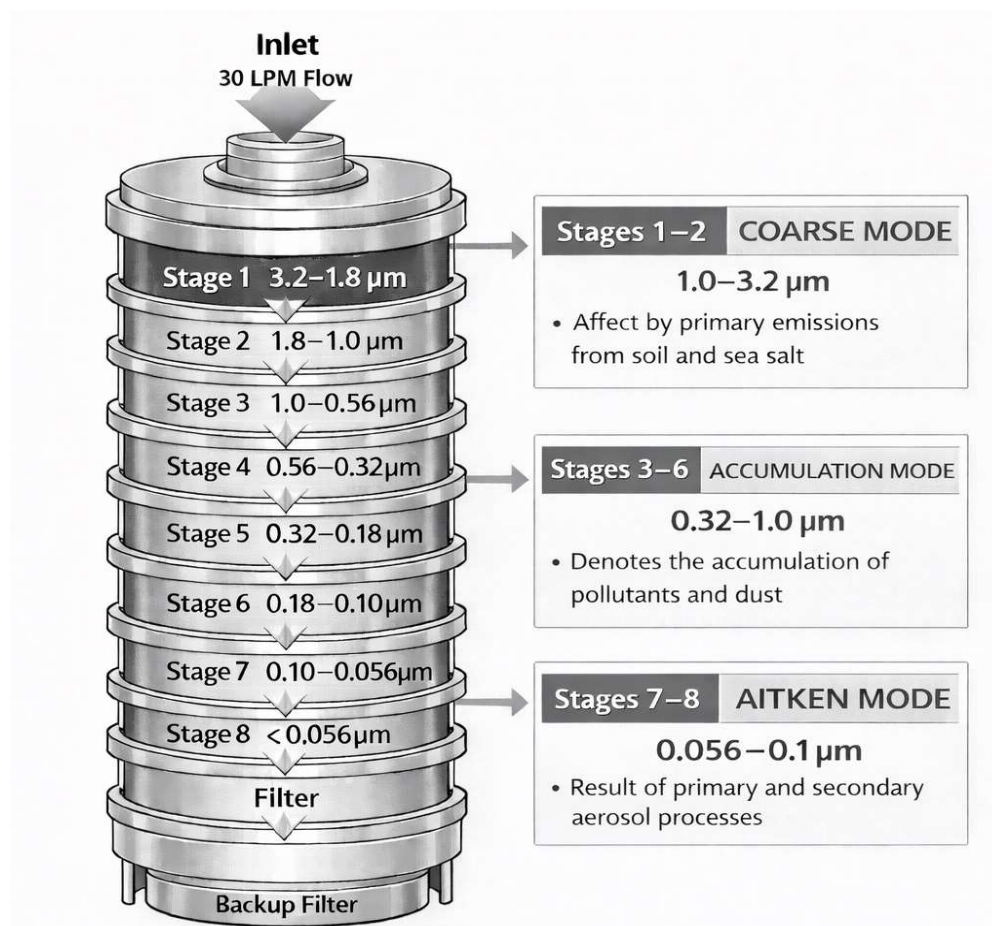


Figure 23. Nano-MOUDI II cascade impactor used for size-resolved aerosol sampling (3.2 μm to <0.056 μm ; 30 L min^{-1}). Size fractions are grouped into coarse (Stages 1–2), accumulation (Stages 3–6) and Aitken modes (Stages 7–8).

Two sets of samples were collected, period 1 (P1) between 12 to 15 January 2023 and period 2 (P2) between 15 to 17 January 2023. Each stage was prepared with a 47 mm PTFE membrane filter, porosity 1 μm (Whatman, Maidstone, UK) and a Si wafer gold-coated (Si/Au wafer) was fixed in the center of the membrane filter. After the sampling each filter plus Si/Au wafer was storage in Petris dish, wrapped in aluminum foil and dropped in inert environments to avoid contamination and protect for damaging before the measurements.

Non-refractory submicron aerosol species online measurements of mass concentration (M_{species}) to organic aerosols (OA), sulfates (SO_4), nitrates (NO_3), ammonium (N_4) and chloride

(Cl⁻) were measured through an Aerosol Chemical Specie Monitor (ACSM, Aerodyne Research Inc., Billerica, USA). The nominal size range of the ACSM ranges from 75 to 650 nm, and the measurements consider the size-integrated (Pöhlker, Mira L. *et al.*, 2018). The measurement of Black Carbon (BC) mass concentration was acquired by a Multiangle Absorption Photometer (MAAP), specific details could be found in (Saturno *et al.*, 2018), each equipment was installed at 325-meter height. The measurements were used to calculate the corresponding mass fractions, f_{species} , of each compound following equation 1:

$$f_{\text{species}} = \frac{M_{\text{species}}}{M_{\text{OA}} + M_{\text{SO}_4} + M_{\text{NH}_4} + M_{\text{NO}_3} + M_{\text{Cl}} + M_{\text{BC}}}$$

Data acquired refers to the average for each P1 and P2, all the masses input is in $\mu\text{g m}^{-3}$.

Particles size distribution is an important parameter, once it defines part of aging and reaction of these particles in the atmosphere (Cai *et al.*, 2020; Colbeck; Lazaridis, 2014). We utilize a Scanning Mobility Particles Sizer (SMPS) model 3082 (TSI Inc., Shoreview, MN, USA) to obtain this information. The air was pumped through a 25 mm diameter stainless steel tube (finetron tubes, Dockweiler AG, Neustadt-Glewe, Germany) and dried to a relative humidity (RH) below 40 % (Franco *et al.*, 2022; Tuch *et al.*, 2009). Particle range cover diameters from 10 to 400 nm and yielded temporal resolution of 30 min, integrated to be adjusted to sampling time of P1 and P2.

Backward particles trajectory

The Hybrid Single-Particle Lagrangian Integrated Trajectory model (HYSPLIT) was applied to the period of sampling to confirm the source contribution to aerosols population. This model is widely used to calculate air masses trajectory (Stein *et al.*, 2015). The calculation of forward and backward trajectory consisting in hybrid methods between Lagrangian approach to calculate the air move and trajectories and Eulerian methodology to consider the airborne particles. In our study the input meteorological data files were obtained from National Oceanic and Atmospheric Administration (NOAA) Global Data Assimilation System (GDAS), the NOAA Global Forecast System (GFS) apply observed data from GDAS into a gridded model space for initializing weather forecasts (Cui; Song; Zhong, 2021; Rolph; Stein; Stunder, 2017).

The HYSPLIT model was run for the period between January 8 and 18, corresponding to a 10-day window, in order to capture wind contributions and potential emission sources influencing the measurements and sample collection. Additionally, particle residence frequency was simulated to estimate how often air parcels passed over the same location. This analysis was conducted over a 5-day period.

Determination of chemical elements

The polycarbonate filters were measured by Energy Dispersive X-ray Fluorescence (EDXRF) the fluorescence equipment (Epsilon 5, PANalytical, Almelo, Netherlands), utilize an Rh X-ray tube with maximum energy of 50 kV voltage targeting Al foil and a spectrometer (EDX-700HS, Shimadzu, Kyoto, Japan) detect the emissions from samples. The configuration setup allows the quantification of 25 chemical elements (Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Cd, Sb and Pb), the limit of detection, detailed calibration and precision of these equipment could be find in (Arana, Andréa Araújo, 2014).

Spectroscopic analysis on size-segregated aerosols particles

The qualitative aspects of Micro-Fourier Transform Infrared Spectroscopy (μ FTIR) are powerful to analyze heterogeneous samples. The association of molecular vibration related with structure-spectral functional groups, especially the organics, make the FTIR technique unique to analyze aerosols (Coates, 2000). We realized μ FTIR measurements in reflection mode using a diffraction-limited IR microscope (Cary 620, Agilent Technologies, Santa Clara, USA) in the IMBUA beamline at the Brazilian Synchrotron Light Laboratory (LNLS), National Center for Research in Energy and Materials (CNPEM). The spectral resolution was configured at 8 cm^{-1} , and range scan from $4000\text{ to }700\text{ cm}^{-1}$, each μ FTIR spectrum was normalized with a reference background spectrum of a clean gold surface, detailed information could be accessed at (Fernando Amato-Lourenço *et al.*, 2024). Thorough comparative analysis with help of FTIR libraries such as KnowItAll IR Spectral Library and NIST (Eds. P.J. Linstrom; Mallard, 2025; Kirchner *et al.*, 2003; Wiley Science Solutions, 2021), the similar compounds in the aerosols samples were identified.

We also performed X-ray Photoelectron Spectroscopy (XPS) measurements in the aerosol samples. This kind of technique has a potential for probe the firsts layers of the material – from 1 to 10 nm of penetration – revealing the chemical active interaction aerosol-atmosphere (Ault; Axson, 2017; Riemer *et al.*, 2019). We could identify and quantify the atomic percentage (At.%), the survey detected elements by XPS were Na, I, O, N, K, C, S, Cl, and Si. The data

were acquired using a PHOBIOS 150 hemispherical analyzer with 9 detection channels (SPECS Surface Nano Analysis GmbH, Berlin, Germany) accoupled in the IPE beamline end station. The beamline setup was adjusted to excitation energy at 1253,6 eV, detector resolution at 40 eV and pass energy at 0.5 eV to measure the Survey Spectra. The synchrotron radiation offered by Sirius allows us to control the excitation energy in the high-resolution spectrum (HRS) to emit the electron from each photoelectron peak 400 eV kinetic energy. The HRS configuration was detector resolution 20 eV and pass energy at 0.1 eV, and spectra shift energy was corrected using a gold standard (99.9% purity). The spectrum analysis utilized Shirley background thus the conductive substrate and a asymmetric line shape with Lorentzian-Gaussian (Song; Peng, 2009). To accomplish the deconvolution of peaks we used reference data as NIST and XPS Database (Thompson *et al.*, 2001; Wagner *et al.*, 1979) and other studies (Guascito *et al.*, 2015; Moulder *et al.*, 1993; Pastuszka *et al.*, 2005; Song; Peng, 2009).

RESULTS AND DISCUSS

Aerosol emission sources detection

The global climate is interconnected through atmospheric currents. These same air currents allow aerosols to travel long distances, transporting various chemical elements across the planet and influencing climatic phenomena (Popovicheva; Ivanov; Vojtisek, 2020; Seinfeld; Pandis, 2006). Analyses using the HYSPLIT model provide evidence of the origin of the aerosols collected during the sampling campaign, as shown in Figure 24.

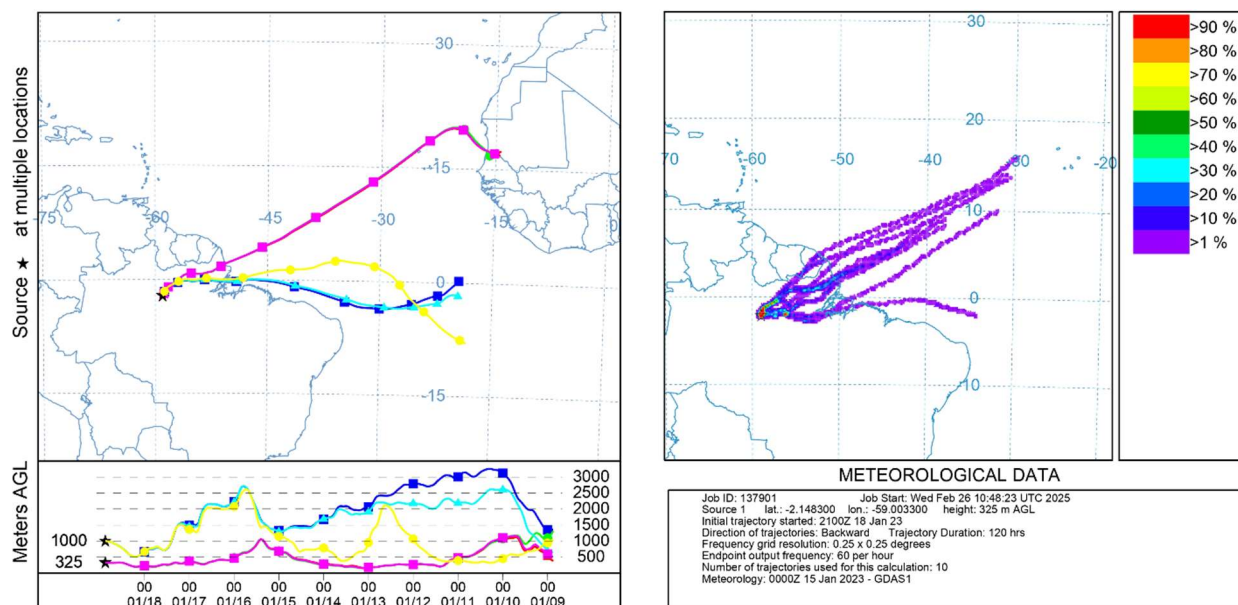


Figure 24. Particle back-trajectories from emission sources were determined using the HYSPLIT model based on GPD data (a) 10-days window calculation between 08 to 18 of January showing the different transport pathways and the vertical displacement profiles and (b) the frequency of particle circulation relative to a give point is displayed.

The back-trajectory calculations were set to a 10-day window starting on 08 January, an extended time window for the trajectory analysis is considered adequate for a more accurate identification of aerosol sources (Franco *et al.*, 2022; Pöhlker, Christopher *et al.*, 2019). This time frame precedes the sampling period and extends through its final day, ensuring that potential long-range emission influences are captured. The analysis reveals three main patterns of aerosol origin, with two trajectories originating from the Atlantic Ocean and one from northern Africa. Therefore, the collected samples are, to some extent, influenced by emissions external to the Amazon region.

Although the aerosols originate from distant regions, as shown in Figure 24a, they tend to decelerate upon reaching the continent. This behavior suggests that, despite their long-range transport, the sampled aerosols are strongly influenced by particles present in the surrounding region, such as biogenic emissions (Artaxo *et al.*, 2022; Guyon *et al.*, 2004; Pöschl *et al.*, 2010). This observation is supported by the trajectory frequency shown in Figure 24b, which was calculated using a 5-day window, the maximum allowed by the HYSPLIT model for this type of analysis. Finally, the analysis also highlights the altitudes at which these particles are transported. Aerosols originating from northern Africa remain below 1000 meters, whereas those from the Atlantic Ocean reach altitudes between 2500 and 3000 meters. These characteristics may be related to particle size and mass. This distinction has physical significance, as dust particles from the Sahara Desert tend to be larger and heavier, while marine

emissions are typically smaller and lighter (Colbeck; Lazaridis, 2014; Rajan *et al.*, 2024; Williams *et al.*, 2024).

Particles size and aerosols composition

Based on the number of collected particles as a function of their diameter, we estimated particle volume and derived the aerosol size distribution (Figure 25). The results indicate that at period 2 (P2), most particles are concentrated in the Aitken mode (50–100 nm), whereas at period 1 (P1) the peak particle number occurs within the 100–300 nm range.

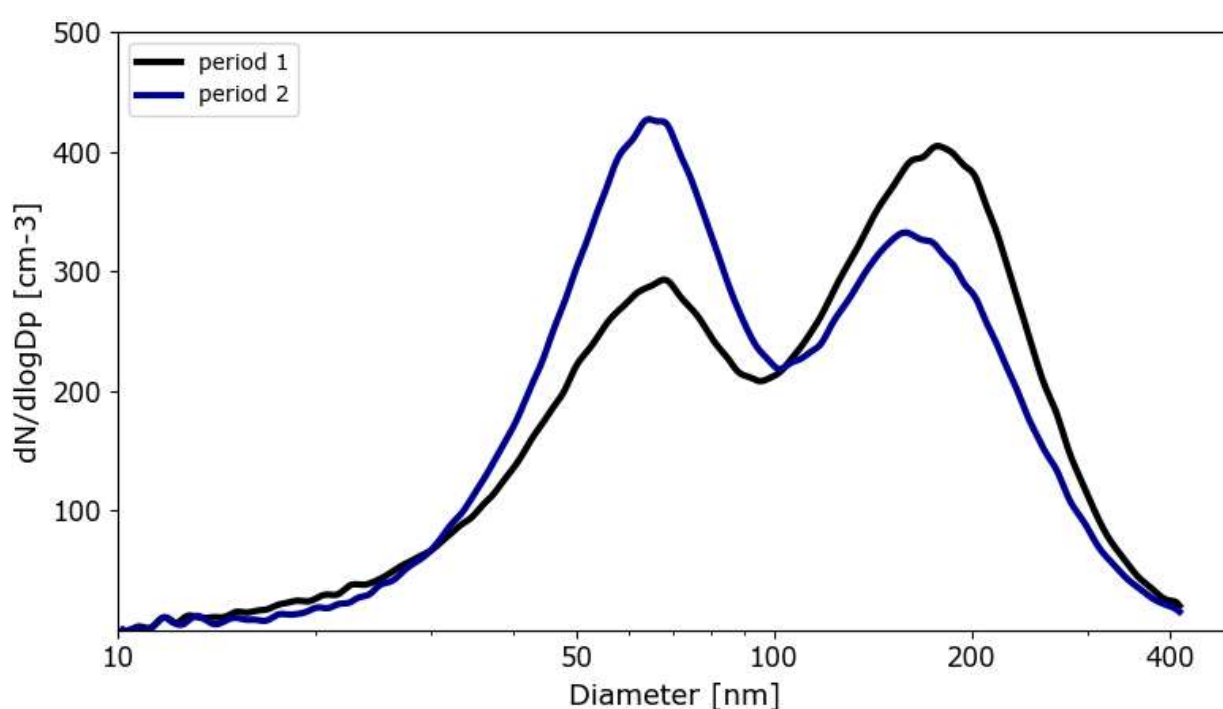


Figure 25. Aerosol particle number distribution as a function of diameter, $dN/d\log D_p$ denotes the particle distribution. Period 1, sampled from January 12 to 15, and period 2, sampled from January 15 to 17, 2023.

The first peak corresponds to the Aitken mode, suggesting that a large fraction of aerosols are in an early stage of development and continue to undergo chemical processing involving secondary organic aerosols (SOA) (Artaxo *et al.*, 2022). A second peak was observed in the accumulation mode, where particles grow in mass through coagulation and condensation processes, leading to significantly longer atmospheric lifetimes, typically ranging from days to weeks (Fröhlich-Nowoisky *et al.*, 2016; Mahilang; Deb; Pervez, 2021; Seinfeld *et al.*, 2016). Machado *et al.* (2024) reported lower aerosol concentrations following rainfall events in the Amazon compared to those observed in the present study. Nevertheless, the mean particle

number concentrations reported here are consistent with typical wet-season values at this site, ranging from 300 to 400 cm⁻³ (Artaxo *et al.*, 2022).

We also highlight the low number concentration of newly formed particles in both periods, represented by aerosols with $D_p < 50$ nm. Additionally, the shift in the Hoppel minimum from ~ 90 nm in P1 to ~ 100 nm in P2 suggests an increase in the effective CCN activation diameter, indicating reduced particle hygroscopicity during the second sampling period. The higher sulfate concentrations within the SOA fraction during P1 (Figure 26) may partly explain the observed differences in particle hygroscopicity. This enhanced hygroscopicity could promote the growth of ultrafine particles ($D_p < 10$ nm) into larger sizes within the Aitken mode. However, these findings are in contrast with those reported by Curtius *et al.* (2024), who observed different growth dynamics to the Amazonian atmosphere.

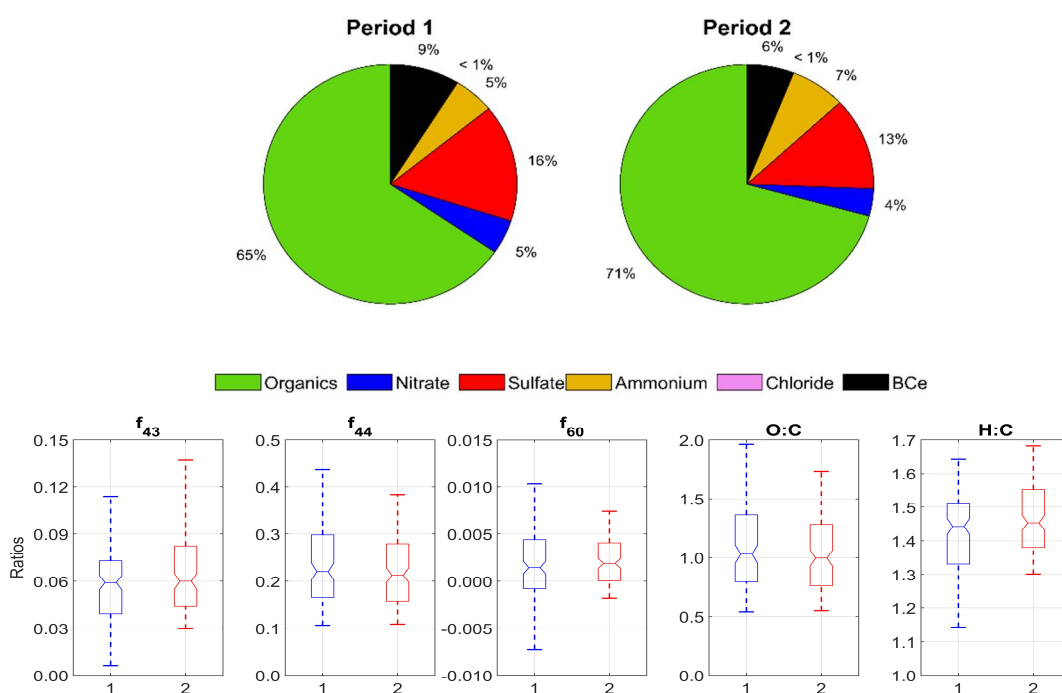


Figure 26. ACSM and MAAP data showing the distribution of compounds in PM₁ aerosol composition across five groups: organics, nitrate, sulfate, ammonium, chloride, and equivalent black carbon (BCe). The O:C and H:C ratios, as well as the organic mass fragments m/z 43, 44, and 60, are presented as boxplots to assess the oxidation state and potential sources of organic aerosols.

It is important to note that the ACSM measures aerosol chemical composition within an aerodynamic diameter range of 75–650 nm, including organic aerosol (OA), which is typically dominated by secondary organic aerosol (SOA). These results can be compared with SMPS and the samples collected between the 4 and 8 stages of the MOUDI (56 – 560 nm). A high concentration of organic compounds was observed in the chemical speciation of aerosols,

accounting for 65% and 71% of the total composition, at P1 and P2, respectively. This is lower than commonly found at this time of year in the Amazon, where the organic fraction typically represents 70 – 90% of the aerosol mass. The signals also suggest the presence of organic compounds containing C–S and C–N bonds, an indication supported by the oxidation processes of gases in the Amazonian atmosphere. These processes, in turn, also favor the activity of CCN (Andreae, Meinrat O. *et al.*, 2018; Artaxo *et al.*, 2022). The inorganic fraction is dominated by sulfate (16% and 13%), ammonium (5% and 7%), nitrate (5% and 4%), while Chloride is lower (<1%). Nevertheless, the results are not close to the typical characteristics of the rainy season in the Amazon under “pristine” conditions (86% organic matter, 6% sulfate, 4% nitrate, and 6% ammonium) (Artaxo *et al.*, 2022),

The elevated sulfate values and the equivalent black carbon (BC_e) concentration reaching 9% and 6% suggest the influence of long-range transport and the contribution of African biomass burning plumes to the aerosol composition (Holanda *et al.*, 2020). By comparing these results with those provided by the SMPS, we can infer that long-range transport predominantly influenced P1. As described by Shen *et al.* (2024), the formation of organic coatings on nearly all primary and secondary particles in the Amazon is an important process. Particles included in the nucleation mode ($D_p < 30$ nm), might be associated with nucleation driven by H₂SO₄, which enhances aerosol growth rates and increase counting number of aerosols inside Aitken mode. On the other hand, primary particles as BC could produce a bulk to aerosols coagulation, inducing a trend of growth process dominated by condensation sinks on pre-existing particles and increasing the number of particles in accumulation mode (Artaxo *et al.*, 2022; Riemer *et al.*, 2019; Tomlin *et al.*, 2021). In this context, it is possible that the atmosphere was saturated, and the high particle load in P1 led to stronger competition for condensable vapors, thereby favoring the accumulation mode. In contrast, during P2, a cleaner atmosphere with lower BC concentrations allowed H₂SO₄ to act more efficiently in particle nucleation, increasing the aerosol number concentration in the Aitken mode.

The organic aerosols were highly oxygenated in both periods, with median O:C ratios ≥ 1 . This degree of oxidation is characteristic of secondary organic aerosol formed predominantly from regional biogenic precursors (Artaxo *et al.*, 2022; Chen *et al.*, 2015). Although the ACSM does not resolve individual molecules, recent high-resolution studies in Amazonia have identified highly oxidized species such as multifunctional carboxylic acids (e.g., MBTCA), isoprene-derived organosulfates (IEPOX-SOA), and monoterpene oxidation products like pinic acid as key contributors to this fraction (Hu *et al.*, 2015; Leppla *et al.*, 2025).

Aliphatic species ($H/C \geq 1.5$; $O/C \leq 0.5$) can be observed, along with aromatic hydrocarbons such as $C_8H_6O_3$, $C_{10}H_{10}O_4$, and $C_{10}H_8O_5$. In addition, unsaturated hydrocarbons with low oxygenation containing N, S, and O (CHON, CHOS, CHONS) and exhibiting high aromaticity ($X_c \geq 2.5$) are also present (Leppla *et al.*, 2025). These findings are consistent with the observed O:C ratios and support the interpretation of a dominantly biogenic, photochemically aged organic aerosol (Thomas *et al.*, 2025).

In ACSM data, different fragments of the organic mass spectrum provide insights into aerosol sources and processing. The relative contribution of m/z 43 is typically associated with less-oxidized, fresh organic aerosol, while m/z 44 is linked to highly oxygenated secondary material. Despite the high variability observed for both mass fragments, the mean values for the two sampling periods were similar, approximately ~ 0.06 for m/z 43 and ~ 0.22 for m/z 44. These results suggest that the organic aerosol composition is predominantly aged and highly oxygenated, consistent with a profile dominated by oxygenated organic aerosol (OOA). The relatively low f_{43} values indicate a limited contribution from fresh hydrocarbon-like organic aerosol (Guo *et al.*, 2020). Therefore, the observed shift in the Hoppel minimum is likely not controlled solely by the oxidation state of the organic fraction, but rather by the combined effects of organic composition, sulfate content, and cloud processing conditions. The fragment at m/z 60 is widely used as a tracer for primary biomass burning emissions, with a threshold of $\sim 0.35\%$ commonly applied to indicate significant influence from fresh smoke (Cai *et al.*, 2020; Cubison *et al.*, 2011). In our case, the fractions of these key markers showed similar patterns across both periods analyzed, suggesting that, from the ACSM perspective, no major differences can be distinguished in the bulk chemical properties of the organic aerosol.

Complementary chemical information was obtained through EDXRF, elemental composition could give important insight along the emissions sources and atmospheric process (Kaushik *et al.*, 2021). The absolute elemental composition of each size fraction (Figure 27) showed an interesting profile, although this information does not directly reflect the internal mixing state of the aerosols this result reinforces the heterogeneity of aerosol composition as a function of particle size.

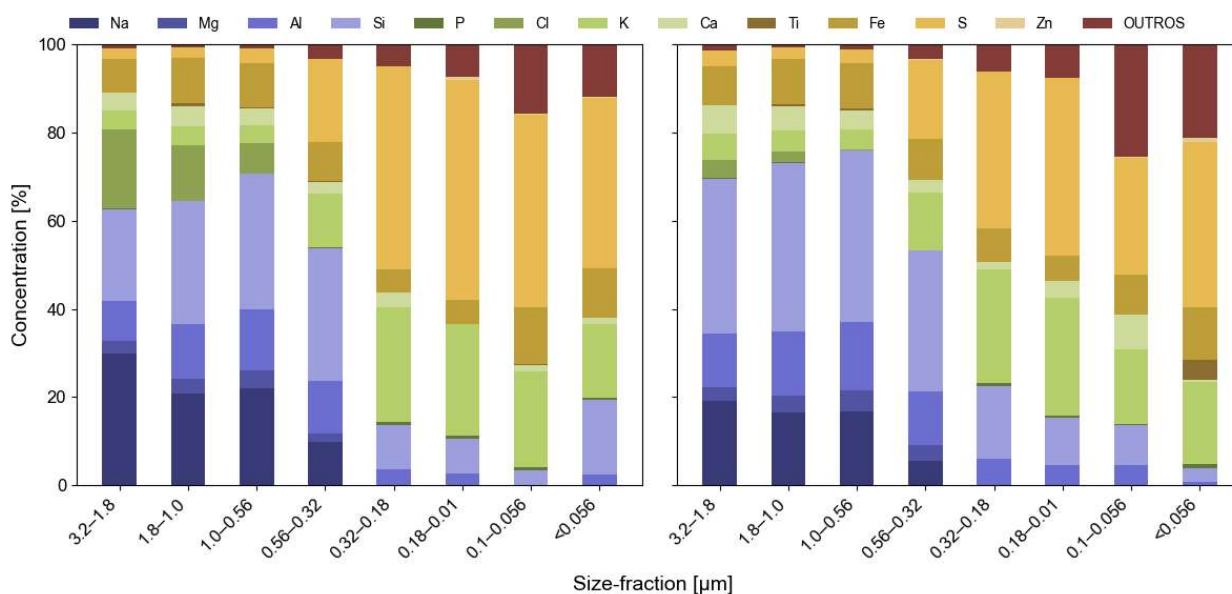


Figure 27. Relative chemical composition to the highest concentrated elements of size-segregated particles from Amazon acquired by EDXRF for period 1 (left) and period 2 (right).

Coarse-mode aerosols exhibit a more heterogeneous elemental distribution of Mg, Ca, K, and Al each show distributions above or nearly 25% in the coarse-mode fractions. The presence of Na, Si and Cl decreases with decreasing D_p , this behavior was expected and the aerosols' source of this fraction can be related with oceanic emissions and dust (Artaxo *et al.*, 1998; Brooks; Thornton, 2018; Colbeck; Lazaridis, 2014; Kaushik *et al.*, 2021). Particles whose diameter ranges between 1.8–1.0 μm show a peak in Ti, this size fraction contains most of the 11 major elements, representing approximately 37% of their total concentration. Sulfur is more abundant in the fine mode, particularly in the 0.56–0.18 μm range, we also observed a high relative concentration to the Zn in particles with $D_p \leq 0.18 \mu\text{m}$. The relative distribution of other elements further indicates that the smallest size range particles are influenced by contributions from both biogenic sources and aged biomass burning (Artaxo *et al.*, 2013; Holanda *et al.*, 2020; Popovicheva; Ivanov; Vojtisek, 2020).

The absolute chemical composition of aerosols also reveals a clear distinction between fine- and coarse-mode particles. Elements such as Na, Mg, and Cl were detected exclusively in particles within the 3.2 – 0.32 μm diameter range. The Na:Cl ratio associated with sea spray was lower than values reported in the literature, where Cl accounts for approximately 55% and Na for 31% (Zieger *et al.*, 2017). The size fraction in which these elements are distributed reflects the growth factor driven by their hygroscopic properties and it also affects the CCN efficacy. The contributions of Si and Al are also higher compared to smaller particles, indicating

the predominance of dust contribution on this size-fraction (Fan *et al.*, 2021; Jianhua *et al.*, 2006).

Although P and Zn are present only in the smaller particles, their overall contribution to aerosol composition remains low ($\leq 1\%$), these elements are “fingerprints” of carbon-rich and organic species from anthropogenic source (Aboh *et al.*, 2009; Cai *et al.*, 2020; Pöhlker, Christopher *et al.*, 2012). The contribution of minor concentrated elements increased with decreasing particle size, reaching approximately 16% in the 0.01 – 0.0056 μm range, with Cd as the main contributor. This heavy metal is not commonly present in the Amazonian atmosphere and may pose health risks. Although its source cannot be precisely identified, Cd is often associated with contamination from mining activities (Garba; Abubakar, 2018). Furthermore, K and S become increasingly dominant in the aerosol composition as particle size decreases, accounting for 60 – 75% of the composition in particles with $D_p \geq 0.32 \mu\text{m}$. Both of elements are signatures of biomass burning in fine particles, their solubility in water could influence the hygroscopicity of the aerosols (Aboh *et al.*, 2009; Viksna *et al.*, 2004). For comparison, the S:K ratio obtained in this study for particles with $D_p \leq 1.8 \mu\text{m}$ was 1.03, whereas values reported for $\text{PM}_{2.5}$ particles were 2.4 in Riga, Latvia, 1.81 in Central Brazil during the dry season, 1.23 in Kwabenya, Ghana, and 2.37 at the same Amazonian site during the wet season (Aboh *et al.*, 2009; Artaxo *et al.*, 2013; Ramos *et al.*, 2024; Viksna *et al.*, 2004).

Overall, the combined analysis of particle size distribution and chemical composition reveals distinct aerosol characteristics between the two sampling periods. P1 is characterized by a greater contribution of accumulation-mode particles and a lower Hoppel minimum ($\sim 90 \text{ nm}$), indicating more efficient CCN activation. In contrast, P2 shows a shift toward the Aitken mode and a higher Hoppel minimum ($\sim 100 \text{ nm}$), suggesting reduced activation efficiency of smaller particles. Although both periods are dominated by highly oxygenated organic aerosol ($f_{44} \approx 0.22$), the similar f_{43} and f_{44} distributions indicate that differences in organic oxidation alone do not fully explain the observed behavior. Instead, the results suggest a combined influence of aerosol composition and atmospheric conditions. In particular, the relatively higher sulfate and equivalent black carbon fractions in P1 point to a modestly stronger anthropogenic influence compared to P2, which may have contributed to enhanced particle hygroscopicity and CCN activation efficiency.

Molecular composition and surface chemical properties

FTIR analysis is particularly valuable in this type of study as it enables the characterization of both organic and inorganic fractions that make up aerosols, different groups

of aerosols such as sulfate, silicates and organics could be at lower troposphere altitudes (Steven F. Maria *et al.*, 2002). Quantitative analysis, however, presents additional challenges due to spectral band overlap and limitations in the use of reference standards for accurate quantification (Courty; Dillner, 2009), because of this, we chose a semi-quantitative approach to evaluate the data. Despite these constraints, the qualitative analysis provided important insights into the variation in aerosol composition as a function of particle size. These results shown in figure 28 clearly demonstrate how particle size is attached with chemical composition, in alignment with the findings from other analytical techniques.

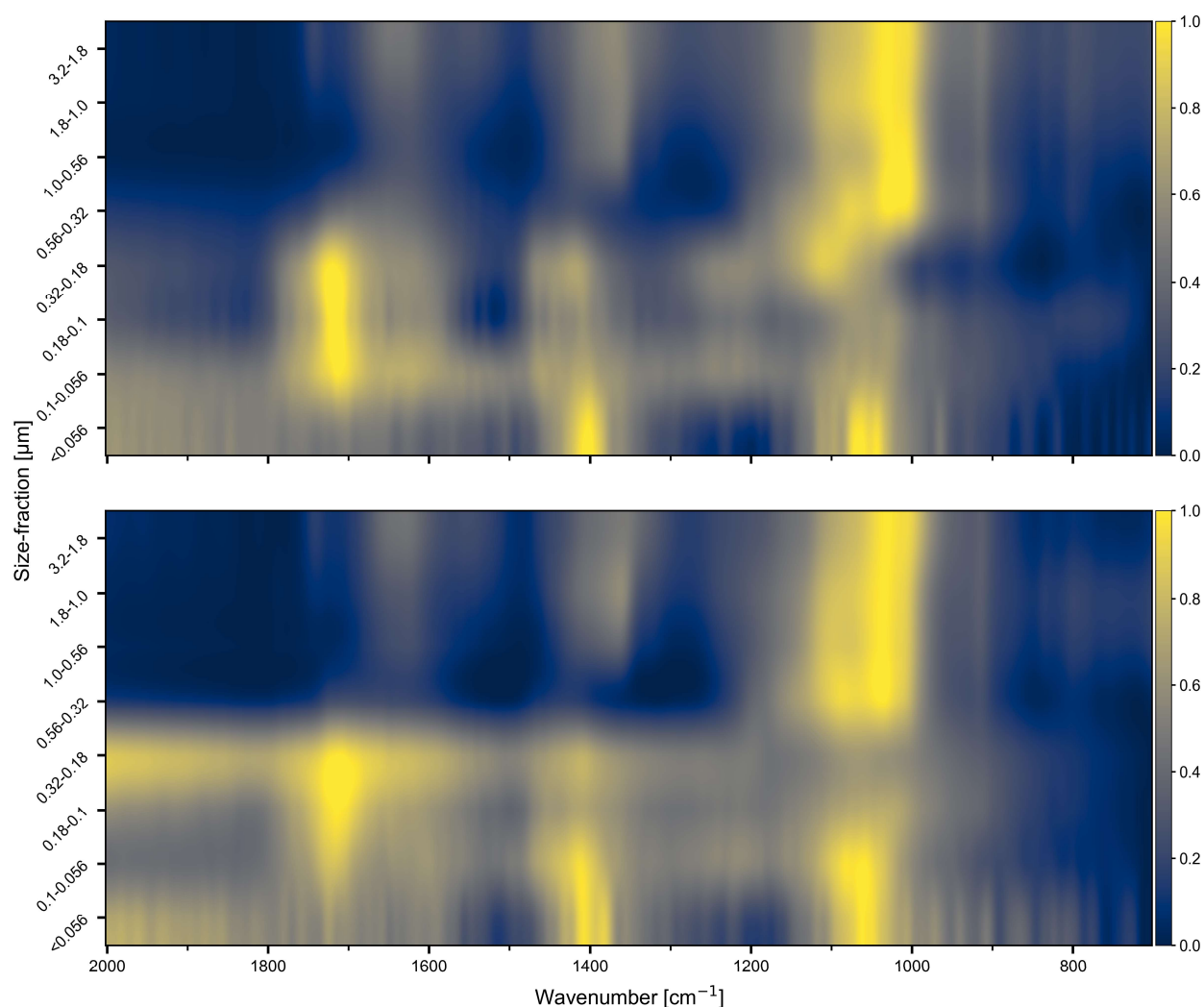


Figure 28. Heatmap of size-segregated aerosol particles based on normalized FTIR spectra from Central Amazon. The intensity of colormap relates to the normalized peak absorbance and indicates the presence of specific molecular groups. The upper panel represents Period 1, while the lower panel corresponds to Period 2.

Carboxylic acid ($\nu\text{C=O}$) around $1700 - 1770 \text{ cm}^{-1}$ and ester functional groups around $1716 - 1742 \text{ cm}^{-1}$ can be overlapped, both are important compounds to the atmospheric acidification process, they are mainly concentrated in small particles ($D_p \geq 0.56 \text{ }\mu\text{m}$) (Courty;

Dillner, 2009; Shankar *et al.*, 2022), with ester also appearing in small amounts in $3.2 - 1.8 \mu\text{m}$. The region between 1650 and 1550 cm^{-1} further confirms the presence of amides, which can be attributed to aliphatic amides (C=O stretching), aromatic amides, as well as N–H and C–H bending vibrations (Shankar *et al.*, 2022), these components are important in the chemical composition of Brown Carbon (BrC) and exhibited more intense peaks in particles with smaller diameters (Laskin; Laskin; Nizkorodov, 2015; Ponczek *et al.*, 2021b).

Compounds in the $1400\text{--}1450 \text{ cm}^{-1}$ region indicate the presence of aliphatic organic material (C–H bending), methylene (CH_2 bending), and methyl groups ($-\text{CH}_3$ bending). The peak intensities point to higher concentrations in the smaller particles, with a noticeable gap in the $0.56\text{--}0.32 \mu\text{m}$ size fraction (Shankar *et al.*, 2022). The compounds present in this region may originate from both biogenic emissions and biomass burning, but also from secondary formation processes (mainly through photo-oxidation) in the presence of precursor gases (e.g., toluene, isoprene) (Artaxo *et al.*, 2022; Coury; Dillner, 2009; Popovicheva; Ivanov; Vojtisek, 2020). Therefore, these compounds may affect the atmosphere in a multifaceted manner, and complementary information would be required for a more in-depth analysis. Other components identified in the FTIR spectra include nitrate ions ($1410\text{--}1385 \text{ cm}^{-1}$), which in the atmosphere can influence aerosol nucleation. Their size-resolved distribution indicates that they are concentrated mainly in the $0.1\text{--}0.056 \mu\text{m}$ fraction. In addition, organonitrates (1280 cm^{-1}) contribute to the organic fraction of aerosols and may form secondarily as products of atmospheric oxidation (Jacobson, M C *et al.*, 2000; Mahilang; Deb; Pervez, 2021), Their presence is also indicative of the complexity of atmospheric chemistry and the aging processes of aerosols (Popovicheva; Ivanov; Vojtisek, 2020).

The spectral region between $1200\text{--}900 \text{ cm}^{-1}$ indicates compounds as sulfates ions (HSO_4^- and SO_4^{2-}) and silicates ($\nu\text{Si=O}$), particularly in the particles size ranging between 3.2 to $0.32 \mu\text{m}$ with a gap of concentration at the size-fraction $0.32 - 0.16 \mu\text{m}$. In the atmosphere the silicates Si–O and O–Si–O at 1035 cm^{-1} and 1000 cm^{-1} respectively, are can directly linked to the dust delivered to the long-range transport, additionally, by comparing the band intensity of both periods, we can also reinforce the greater influence of African emissions in the period 1. They also have an important on the mixture state specially when associated with humic-like organic matter, these complex chemical environment affect directly the radiative balance (Aboh *et al.*, 2009; Gaffney; Marley; Smith, 2015).

The differences in organic and inorganic composition between Periods 1 and 2 are subtle, yet they reveal small but meaningful shifts in the relative abundance of key compounds that may be critical for understanding aerosol aging and particle growth processes in the

Amazon region. Both periods exhibit trace components indicative of highly oxidized particles in the $D_p > 0.32 \mu\text{m}$ size range, consistent with ACSM observations and providing further evidence of size-dependent chemical heterogeneity. Minor variations in oxygenated organic fractions, together with the presence of sulfates and silicates, suggest potential effects on water uptake and influence particle growth dynamics. These findings are consistent with the observed differences in the Hoppel minimum observed in P1 and indicate that subtle differences in composition and mixing state, including a modest anthropogenic contribution, appear to contribute in modulating CCN activation efficiency.

These results reinforce the importance of submicron aerosols and highlight compositional differences between the Aitken and accumulation modes. In particular, particles in the $0.32\text{--}0.56 \mu\text{m}$ size range are of special interest, as they represent a transitional regime where multiple compounds—such as carboxylic acids, aliphatic organics, sulfates, and silicates—coexist.

Surface chemical information is among the most critical and challenging properties to characterize in atmospheric aerosols. The aerosol surface acts as a key interface where water vapor condenses, promoting particle growth, and where gas-phase uptake and secondary chemical reactions occur. These processes directly influence cloud microphysics and, consequently, the macroscopic properties of clouds. Furthermore, surface composition provides valuable insights into particle mixing state and chemical heterogeneity (Seinfeld *et al.*, 2016; Tomlin *et al.*, 2021). To our knowledge, this study represents the first to characterize these surface properties for Amazonian aerosols, providing novel insights into the physicochemical properties of particles that play a crucial role in regulating regional and global climate. The XPS analysis demonstrates singular results to the size-segregated aerosols, data showed the presence of 9 elements in the surface of particles. The active chemical surface prominently features a smaller subset of elements, including Na, I, O, N, K, C, S, Cl, and Si. The analysis of high-resolution peaks of the chemical elements was used to determine the relative concentration of each element in the aerosol surface composition. Figure 29 shows the normalized carbon and sodium peaks for different size-fractions. The intensity of the peaks in the high-resolution spectrum gives information about the concentration of the chemical element analyzed in the high-resolution spectrum (Major *et al.*, 2020). It is also possible to observe the presence of shoulders associated with each peak, suggesting the existence of chemical species.

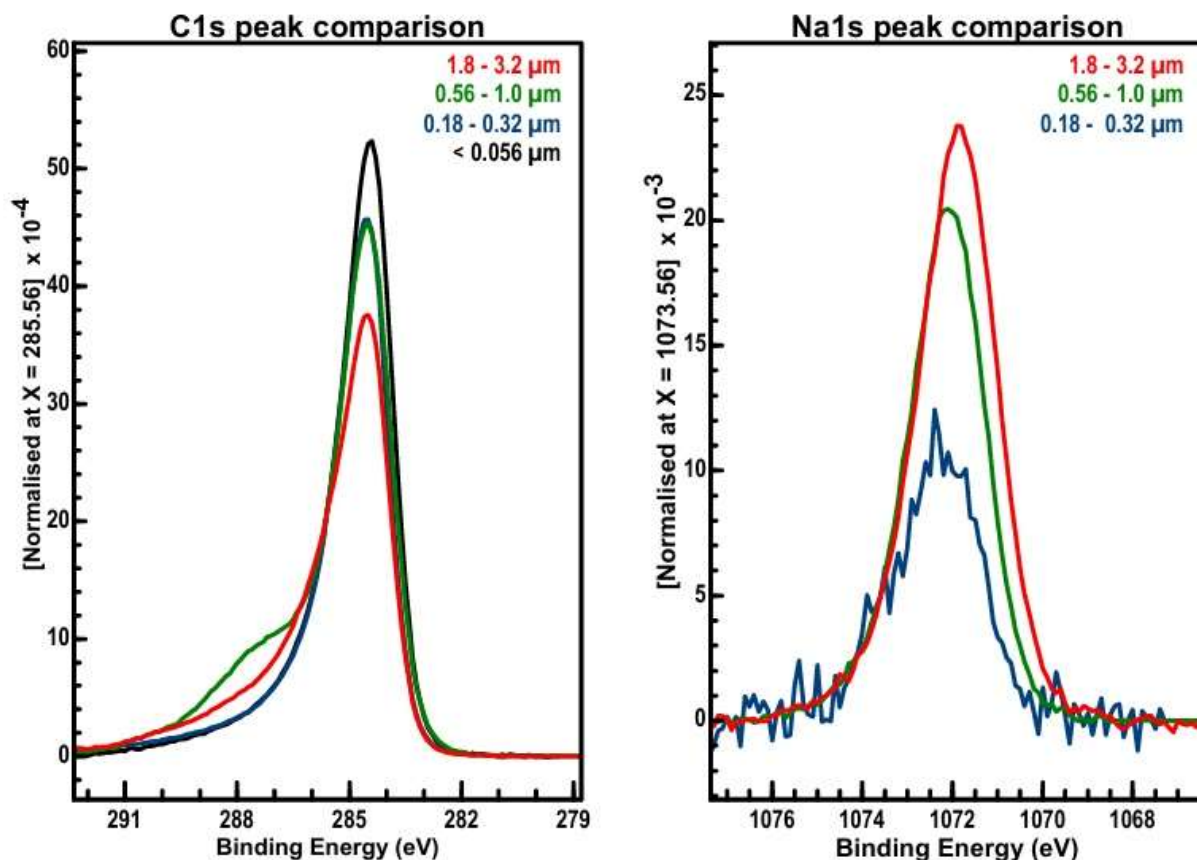


Figure 29. Photoelectron peaks to C1s and Na1s normalized, showing the differences of concentration and curve shape for size-segregated aerosol particles from Central Amazon.

One of the key findings was that Na and Cl were absent in the surface of smaller particles, while the larger particles show a significant contribution of Na reaching a relative maximum concentration of 9.06% at the fraction with diameter between 1.0–0.56 μm. It indicates that oceanic emissions predominantly affect the surface's composition of larger particles, although their impact on hygroscopicity is still subject to uncertainty (Kaushik *et al.*, 2021; Zieger *et al.*, 2017). The results concerning relative atomic concentrations are presented in Figure 30. Carbon and oxygen concentrations vary significantly across particle size fractions. Carbon is predominantly enriched in the finer aerosol fractions, whereas oxygen shows higher concentrations in larger particles. Nitrogen does not show significant variability across the size ranges. Sulfur concentrations are higher in the smallest particles, particularly those with $D_p < 0.056 \mu\text{m}$, consistent with observations from EDXRF measurements. Furthermore, the surface composition profile suggests that the enrichment of sulfur in smaller particles may be associated with particle growth, while increasing oxidation levels are observed as particles evolve toward larger diameters.

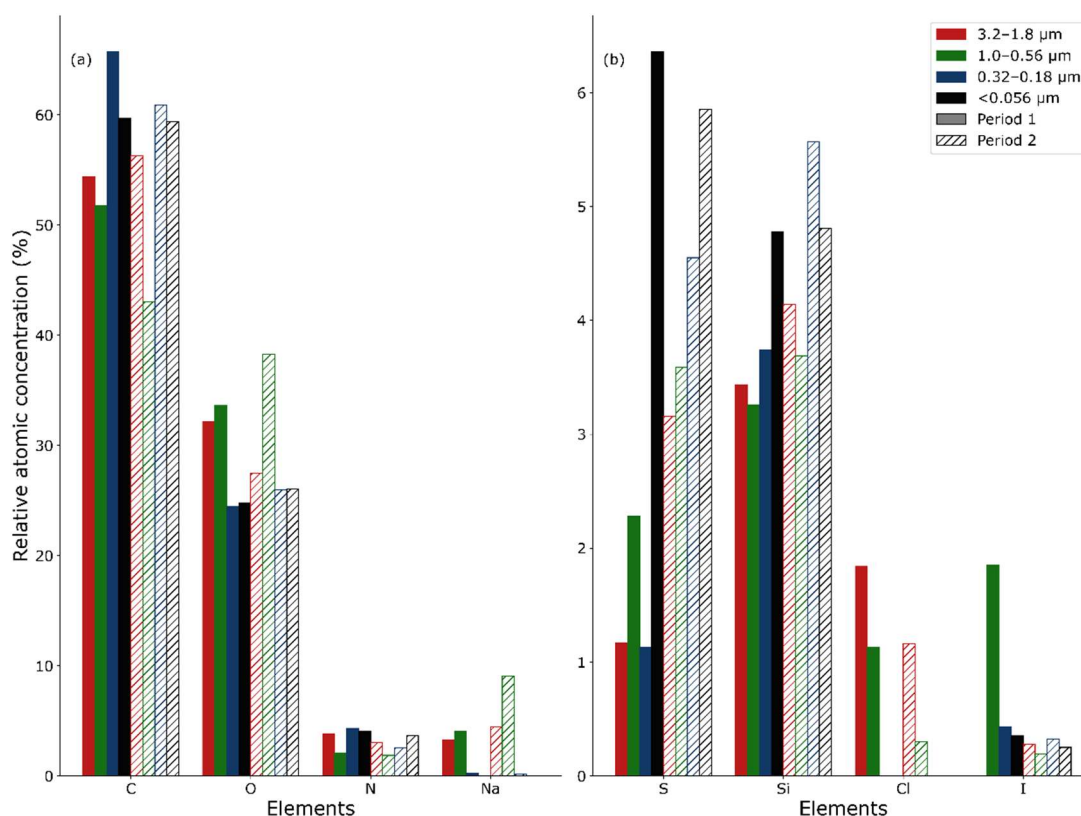


Figure 30. Relative atomic concentration at chemical active surface composition of aerosol particles from Amazon for different size-fractions measured through XPS.

The chemical environment of the carbonaceous component at the aerosol surface was investigated through C1s peak fitting, revealing that the concentration of different chemical bonds varied with particle size (Table 1). The O/C ratio at the surface of larger particles exhibited significant variability, suggesting heterogeneity in the oxidation state. Despite this variation, oxidation tended to be higher in intermediate diameter ranges, particularly in the 1.0–0.56 μm fraction, which reached a value of 0.89 in period 2. For smaller particles, the O/C ratios were relatively consistent and remained at low values. Across all size fractions, the N/C ratio ranged from 0.04 to 0.07, indicating only minor differences in relative nitrogen concentration as a function of particle size. The component assigned to C–O/C–N showed substantial variability among particle sizes and between the two sets. Considering the N/C ratio, we can infer that nitrogen contributions are relatively similar across all samples, and that the main source of variation arises from the C–O group.

Table 3. Distribution of carbon functional groups in aerosols from the central Amazon, obtained through peak fitting of the high-resolution C 1s spectra for different size ranges. Samples are named by [period_size range].

Sample	Ratio O/C	Ratio N/C	C=C	C-C/C-H	C-O/C-N	C=O	Carboxyl	Carbonate
P1_3.2-1.8	0.59	0.07	49.49	31.72	10.33	5.47	1.92	1.06
P1_1.0-0.56	0.65	0.04	57.46	20.03	9.17	9.94	2.59	0.82
P1_0.32-0.18	0.37	0.07	53.41	35.14	5.77	2.81	1.84	1.02
P1_0.056	0.42	0.07	66.70	23.51	6.03	2.48	0.92	0.35
P2_3.2-1.8	0.49	0.05	56.89	37.06	3.19	1.14	0.91	0.80
P2_1.0-0.56	0.89	0.04	59.24	28.17	6.24	3.39	1.95	1.00
P2_0.32-0.18	0.43	0.04	76.12	17.22	4.62	1.31	0.62	0.11
P2_0.056	0.44	0.06	68.03	19.70	8.00	2.87	0.88	0.52

The concentrations of C=C bonds range from 76.12% to 49.49%, while C–C/C–H bonds vary between 37.06% and 17.22%, whereas smaller particles are dominated by aromatic carbon (C=C), indicating a lower degree of oxidation and reduced polarity. Oxygenated species such as carbonyl (C=O) and carboxyl (COOH) groups tend to occur in higher concentrations in particles of intermediate diameters. The enrichment of carboxylic acid functional groups in the particulate phase, driven by their low volatility, serves as a marker of atmospheric processing and the transition toward multigenerational oxidation states of organic aerosols (Russell; Bahadur; Ziemann, 2011). This distribution suggests that particles in the accumulation mode have undergone more extensive atmospheric processing, potentially including cloud processing, leading to enhanced surface oxidation and increased hygroscopicity (Artaxo *et al.*, 2022; Lee *et al.*, 2020). These findings are consistent with the ACSM-derived high f44 values and support the interpretation that CCN activation efficiency is strongly influenced by size-dependent chemical composition. This supports the interpretation that a higher contribution of secondary and surface-processed organic material. Despite the higher oxidation levels observed in P2 for certain size fractions, the overall shift in the Hoppel minimum toward larger diameters suggests that factors beyond oxidation, such as inorganic content and mixing state, play a critical role in modulating cloud activation processes. The detection of carbonates is also noteworthy, their decreasing abundance with decreasing particle size is consistent (Tuyishime *et al.*, 2025), as this inorganic carbon is typically associated with soil resuspension, thus contributing predominantly to the coarser fractions.

These results suggest that particles may exhibit a core–shell structure, consistent with a BC-rich core—typically composed of aromatic carbon—with a surrounding shell of processed

organic material with varying degrees of oxidation. The combined dataset indicates the presence of a chemical gradient, whereby smaller particles show higher carbon content, lower oxygenation, and reduced hygroscopicity, while particles in the intermediate size range exhibit more oxygenated surfaces and enhanced CCN activation efficiency. Moreover, a size-dependent oxidation gradient appears to play an important role in particle growth and CCN activation (Andreae, Meinrat O. *et al.*, 2018).

CONCLUSIONS

This study provides multiscale advanced characterization of lower tropospheric aerosols in the central Amazon during the wet season, integrating both in situ and laboratory analyses. This approach enables the integration of physicochemical information at a level not previously reported for the region. The results reveal a clear size-dependent chemical gradient, in which smaller particles are enriched in aromatic carbon and exhibit lower oxidation levels, whereas particles in the accumulation mode display enhanced surface oxidation and a higher abundance of oxygenated functional groups. These findings indicate that intermediate particle sizes represent a key regime of atmospheric processing, likely associated with aging and cloud-related transformations.

Despite both sampling periods being dominated by highly oxygenated organic aerosol, subtle differences in chemical composition were observed. Period 1 exhibited relatively higher sulfate and equivalent BC contributions, suggesting a modestly stronger influence of long-range transported anthropogenic emissions, whereas Period 2 was more organic-dominated. These compositional variations are consistent with the observed shift in the Hoppel minimum from ~90 nm to ~100 nm, indicating differences in CCN activation efficiency between the two periods. Nevertheless, dynamic atmospheric conditions and even minor contributions from long-range anthropogenic transport may induce substantial changes in the particle population.

The integration of bulk and surface-sensitive techniques highlights the importance of particle surface chemistry and mixing state in controlling aerosol hygroscopicity and cloud activation. In particular, the XPS results suggest that surface composition does not necessarily reflect bulk composition, reinforcing the need to consider surface-specific properties when evaluating aerosol–cloud interactions. High-resolution C 1s spectra further highlighted differences in the quantity and types of carbon present on the particle surfaces. Larger particles

exhibited more chemically complex surfaces compared to smaller ones, which can directly influence the hygroscopic properties of the particles.

In summary, our data indicate that (1) the high particle load in the accumulation mode generates competition for condensable vapors, potentially favoring growth of particles within this same mode, and (2) the 0.56–0.32 and 0.32–0.16 μm size fractions are particularly relevant for understanding aerosol formation and physicochemical properties, as they represent a clear transition zone in terms of chemical composition and particle processing. Moreover, these results (3) advance the microphysical understanding of Amazonian aerosols by combining a comprehensive set of in-situ and laboratory-based measurements, and (4) provide a comprehensive characterization of the distinct features of aerosol size fractions, serving as a valuable reference for future studies aimed at investigating specific processes associated with each fraction.

Overall, the results demonstrate that even subtle variations in aerosol composition and mixing state can modulate particle growth and CCN activation in the Amazonian atmosphere. These findings contribute to a more detailed understanding of aerosol–cloud–climate interactions in a region that plays a critical role in the Earth system and emphasize the importance of multi-technique approaches for advancing atmospheric aerosol research.

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5. MEASUREMENTS OF ATMOSPHERIC AEROSOLS THROUGH SYNCHROTRON X-RAY IMAGING

The studies presented in this chapter derive from 6-months international PhD research completed in May 2025 at the MAX IV Laboratory, Sweden, involving X-ray imaging experiments and significant technical development in synchrotron methods and sample preparation for atmospheric aerosols.

INTRODUCTION

Natural aerosols emissions play a unique influence in climate dynamics, directly affecting the solar radiation balance on the Earth's surface and indirectly shaping cloud formation properties. This emphasizes the central role of aerosols in climate change research, positioning air pollution as a critical aspect of ongoing scientific and policy discussions. Precipitation depends strongly on the concentration of cloud condensation nuclei (CCN), which provides surfaces for water vapor to condense into droplets. However, the chemical composition and size of these particles can alter the characteristics of clouds. For instance, a higher particle concentration does not necessarily increase precipitation, particularly when particles are small and hygroscopic, as is often the case of anthropogenic emissions, which include higher levels of soot and sulfates, primarily in the fine aerosol fraction (Santos, L. F.E.D. *et al.*, 2024).

Aerosols sources shift between the wet season (November–February) and dry season (March–October), impacting the chemical composition and shape of particles. During the wet season, biogenic particles and primary organic aerosols dominate the smallest particles size fraction in forest emissions and are highly effective as CCN, thus supporting efficient precipitation. In contrast, biomass burning emissions introduce smaller, hygroscopic particles that change cloud microphysics, suppressing precipitation and prolong droughts through mechanisms that are still not fully quantified (Bzdek; Reid; Cotterell, 2020; Malhi *et al.*, 2008). Understanding these dynamics is critical to bridging significant gaps in aerosol–cloud interactions, particularly in regions like the Amazon, where aerosols directly influence regional hydrology and the global climate. During the dry season complex microaggregates formed by pyrolysis, containing Brown Carbon (BrC), Black Carbon (BC), soil particles, and secondary atmospheric compounds are emitted. Real-world particles are generated by heterogeneous and irregular process that differ fundamentally from the controlled conditions produced in the

laboratory, studying these particles results in more accurate representation of aerosols nature (Ault; Axson, 2017). Moreover, understanding the mixing state of microaggregates of environmental sources and their role in aerosol aging process is essential to refine climate models and improve predictions of aerosol-climate interactions. These findings may also support more effective approaches related to climate change mitigation.

MATERIAL AND METHODS

The Pantanal is one of the five major biomes in Brazil and is located near the Geodesic Center of South America. The climate is classified as Tropical Wet, with rainy summers and dry winters, *Aw* in the Köppen–Geiger classification. Climatic factors, such as the dynamics of seasonal rainfall with three distinct phases—flood, drought, and ebb—together with its flat relief, favor periodic inundation of parts of the biome, making it one of the largest floodplains in the world. These unique characteristics, combined with its endemic fauna and flora, led UNESCO to designate the Pantanal as a World Heritage Site and a Biosphere Reserve (Filho *et al.*, 2014; Köppen; Geiger, 1936; Nobre; Sampaio; Salazar, 2007). Climate change is becoming increasingly intense in the Pantanal and Amazon, altering both the hydrological and carbon cycles in these regions (Malhi *et al.*, 2008), which are closely interconnected through atmospheric circulation. Land-use changes, driven by the expansion of agricultural areas and gold mining, have modified the aerosol population in the region, particularly during the dry season, which is characterized by a mixture of Black Carbon (BC) and dust (Ramos *et al.*, 2024). Samples were collected at the Advanced Research Base of the Pantanal (BAPP), 16°29'56'' S, 56°24'47'' W, in September 2025, coinciding with one of the most severe fire episodes in history.

Sample preparation followed a novel procedure in which aerosols were extracted by sonication in deionized water, subsequently dried, and embedded in two types of resin (Spurr-like and TTE:MMHA). All samples were sectioned by microtomy into 100 nm slices and deposited onto two types of substrates: 400-mesh copper TEM grids and silicon nitride (Si₃N₄) windows. For nano-XRF measurements, an excitation energy of 8.8 keV was used to identify elements associated with soil resuspension and biomass burning. For STXM measurements, image stacks were acquired at the C K-edge and N K-edge to probe the nanoscale chemical composition.

Five image stacks were acquired at the C K-edge and three at the N K-edge at the same locations. Measurements at the O K-edge were discarded due to acquisition issues, and a problem with I_0 measurement further affected data quality for STXM experiments. A total of 15 individual particles and aggregates were analyzed by nano-XRF, radiation damage from the beam occasionally caused particle displacement and data loss. To mitigate these effects, parameters such as beam intensity and dwell time were adjusted during acquisition, and these modifications were subsequently considered in data analysis. All measurements were automated via dedicated scripts, enabling more efficient use of beamtime, and no issues were encountered with the automation process. We used multiple types of software to conduct data analysis including aXis2000, PyMca, Orange, Athena and Fiji.

RESULTS

STXM measurements at the C K-edge in the 280–300 eV range were converted to optical density, as shown in Figure 31a. A cluster analysis revealed three distinct regions characterized by different internal chemical patterns. These regions are spatially distributed from the particle edge toward the core, which is noteworthy as it suggests that different atmospheric processing mechanisms may occur within different parts of the same particle. The spectra associated with the regions of interest (ROIs) were compared with reference spectra of soot (Obst *et al.*, 2011) and the embedding spurlike resin used during sample preparation (Falcones *et al.*, 2025). Although the analyzed feature corresponds to a small aerosol agglomerate resulting from sample preparation, the spectral variability across the ROIs indicates significant chemical heterogeneity at the submicron scale.

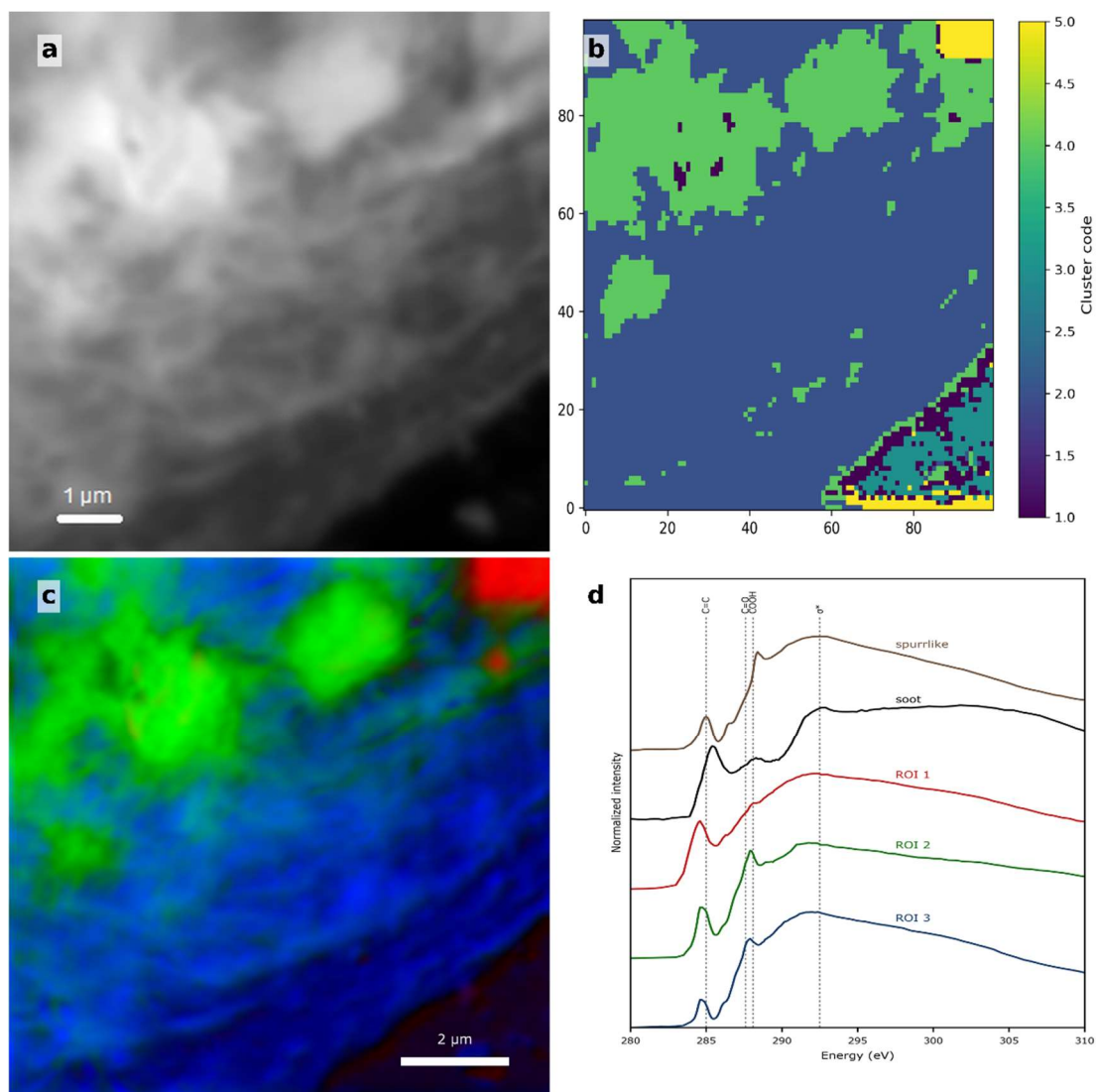


Figure 31. (a) Average STXM image of the C K-edge stack (280–310 eV). (b) Principal component analysis (PCA) combined with cluster analysis to identify regions of interest exhibiting significant chemical variability. (c) RGB composite of the regions identified in (b). (d) Comparison of C K-edge NEXAFS spectra of the substrate resin (Spurr-like), soot, and three regions of interest, calibrated using an internal standard.

The spectral sequence from ROI 1 to ROI 3 suggests a progressive change in the relative contribution of aromatic and oxygenated carbon within the agglomerate, indicating that carbonaceous components are not uniformly distributed. Variations in the pre-edge feature around ~ 285 eV ($\text{C}=\text{C}$ π^*) and the absorption band near ~ 288 eV ($\text{C}=\text{O}$ and COOH) reveal differences in the degree of oxidation across the particle. A relative shift of the pre-edge peak toward higher energies is indicative of a higher oxidation state and is predominantly observed in the particle core, where the presence of oxygenated functional groups further supports this interpretation (Fraund *et al.*, 2019; Lanzirotti; Tappero; Schulze, 2010; Obst *et al.*, 2019). In

contrast, the particle edge shows no significant pre-edge shift and is dominated by less oxidized carbon, with a stronger contribution from aromatic and aliphatic components. This distribution suggests the presence of an internal chemical gradient within the agglomerate, likely reflecting differences in exposure to atmospheric aging processes (Yuan; Zhao, 2023)

As these samples were collected directly from the environment in a remote region of the Brazilian Pantanal during the dry season of 2025, the presence of particles influenced by biomass burning is plausible, given the well-documented increase in fire activity during this period (NASA, 2026). In this context, the nano-XRF results provide key insights into the source attribution and chemical variability of individual aerosol particles. As shown in Table 4, a high degree of compositional variability is observed among individual particles, reflecting the complexity of the atmospheric aerosol population. Elements typically associated with mineral dust, such as Si, Al, Fe, and Ti, are particularly enriched in larger particles (e.g., Scans 11 and 27), suggesting contributions from soil resuspension (Garba; Abubakar, 2018; Holanda *et al.*, 2023; Ramos *et al.*, 2024). In contrast, elevated concentrations of K, S, Cl, and P, especially in smaller particles such as Scan 18, are consistent with emissions from biomass burning, which is expected to be a dominant regional source during the dry season in the Pantanal. In several cases (e.g., Scans 11 and 13), the coexistence of elements associated with both mineral dust and biomass burning indicates the presence of mixed particles, suggesting aggregation processes or atmospheric aging (Ponczek *et al.*, 2021a; Riemer *et al.*, 2019; Saturno *et al.*, 2018). This observation highlights the importance of mixing state in determining aerosol properties.

By comparing elemental composition with particle areas, a clear size-dependent trend emerges. Larger particles tend to be dominated by mineral components, while smaller particles exhibit higher relative contributions from biomass burning tracers. Furthermore, nano-XRF elemental maps of individual particles, obtained with a spatial resolution of approximately 100 nm, provide detailed insight into intra-particle heterogeneity (Figure 32). The spatial distribution of key elements such as Cl, S, and Ca reveals non-uniform internal structures, indicating that these components are not homogeneously mixed but instead form localized domains within individual particles.

Table 4. Particle area and elemental mass fractions of aerosol samples collected in the Pantanal, providing an integrated overview of size and compositional variability.

Elements	scan 10	scan 11	scan 13	scan 18	scan 21	scan 27
Na	0.0159	0.0691	0.0196	0.0360	0.0185	0.0166
Mg	0.0081	0.1307	0.0868	0.0001	0.0000	0.0112
Al	0.0325	0.1283	0.0547	0.0527	0.0236	0.0410
Si	0.0262	0.2189	0.0639	0.0214	0.0088	0.1387
P	0.0013	0.0195	0.0215	0.0101	0.0051	0.0070
S	0.0749	0.6262	0.8587	0.5464	0.2848	0.0221
Cl	0.1287	1.4900	0.4252	2.1400	1.3280	0.2290
K	0.0178	0.1030	0.2689	2.8800	1.2320	0.0043
Ca	0.0464	0.5079	0.0678	0.4583	0.1750	0.0046
Ti	0.0510	0.4766	0.0215	0.0466	0.0070	0.0031
Cr	0.0095	0.0933	0.0139	0.0065	0.0057	0.0009
Mn	0.0016	0.0087	0.0052	0.0055	0.0031	0.0009
Fe	0.0548	0.5722	0.1561	0.0804	0.0880	0.0057
Ni	0.0122	0.0803	0.0229	0.0071	0.0043	0.0009
Particle area (μm^2)	362	2.97×10^4	3.27×10^3	81	4.92×10^3	1.95×10^4

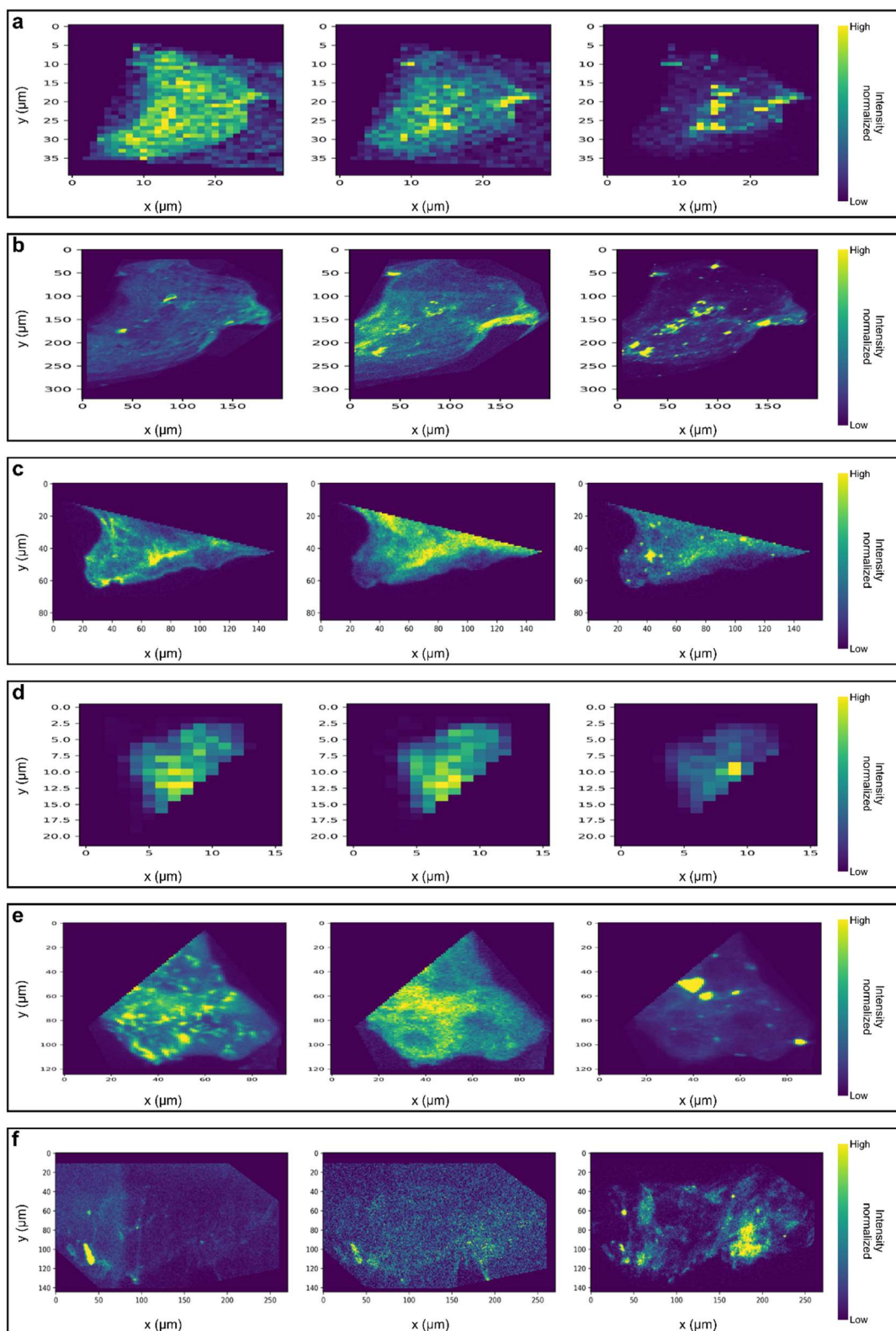


Figure 32. Panel showing X-ray fluorescence (XRF) maps of Cl, S, and Ca for scans (a) 10, (b) 11, (c) 13, (d) 18, (e) 21, and (f) 27. Maps are presented in normalized intensity and reveal spatial heterogeneity in elemental distribution across the analyzed regions.

Building on these observations, the spatial correlations between elemental concentrations provide further insight into particle composition and intra-particle heterogeneity. As each scan corresponds to an individual aerosol particle, the variability in correlation strength across scans indicates that the association between Cl, S, and Ca differs from particle to particle, reflecting distinct compositions and mixing states. Scan 18 exhibits consistently strong correlations across all elemental pairs (Cl–S, Cl–Ca, and S–Ca), indicating a well-defined spatial co-distribution of these elements within the particle. This behavior suggests a relatively homogeneous internal structure, where these species are closely associated at the submicron scale (Zhang, Leiming *et al.*, 2001). In the context of samples collected in the Pantanal during the dry season, such co-occurrence may be consistent with particles influenced by biomass burning, where potassium, sulfur, and chlorine are commonly observed together and may be internally mixed through atmospheric processing (Li *et al.*, 2021).

More selective associations are observed in other particles. For example, Scan 21 shows stronger correlations for Cl–Ca and S–Ca, while the Cl–S relationship is comparatively weaker, suggesting that calcium-bearing phases may be more closely associated with sulfur and chlorine within specific regions of the particle. This behavior may indicate interactions between mineral components and secondary species, potentially reflecting heterogeneous processing. Similarly, Scan 13 presents a stronger correlation between S and Ca, suggesting localized co-occurrence of these elements, which may be associated with secondary sulfate formation on mineral surfaces or mixed-source contributions (Russell; Bahadur; Ziemann, 2011).

These variations highlight differences in internal chemical organization between individual particles, ranging from more homogeneous distributions to clearly heterogeneous structures with distinct elemental domains. Taken together with the compositional trends observed in Table 4, these results suggest that the aerosol population likely includes particles dominated by biomass burning signatures, particles influenced by mineral dust, and particles

resulting from the mixing of these sources during atmospheric transport (Georgoulas *et al.*, 2016; Holanda *et al.*, 2020; Ramos *et al.*, 2024).

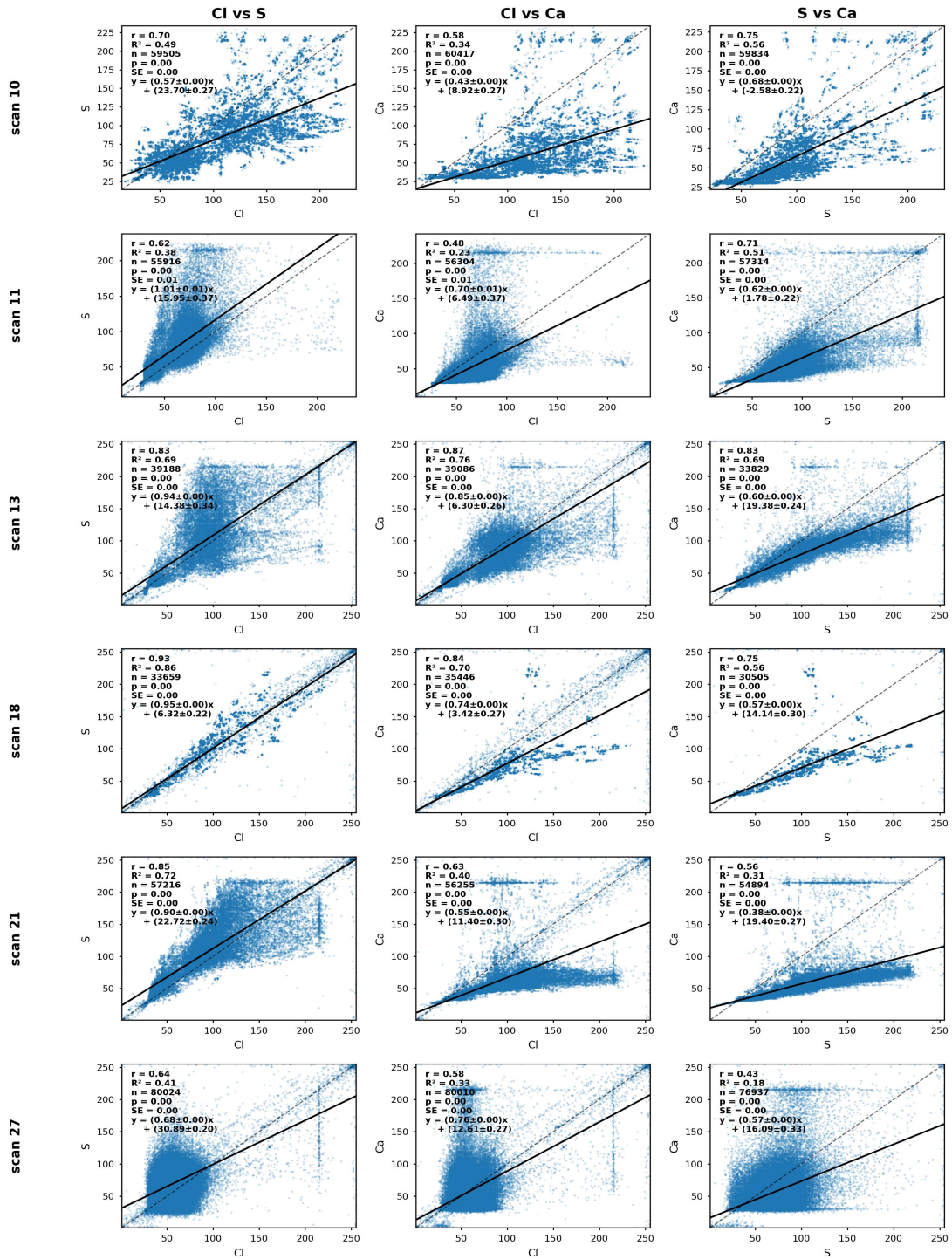


Figure 33. Pixel-by-pixel correlation plots between Cl, S, and Ca for scans 10, 11, 13, 18, 21, and 27. Pearson correlation coefficients (r), coefficients of determination (R^2), p -values, and standard errors (SE) are reported for each case. The results reveal variable degrees of elemental association across scans, suggesting differences in particle composition and mixing state.

Overall, the results indicate that source mixing and aging influence particle composition at the single-particle level, with evidence of both relatively homogeneous particles and more heterogeneous, internally structured aggregates. In addition, ongoing work focuses on developing a correlative methodology to align STXM and nano-XRF images, enabling the integration of chemical speciation and elemental mapping within the same region of interest. This approach is expected to provide further insight into the relationship between carbonaceous functional groups and elemental composition, advancing the understanding of aerosol formation and transformation processes.

CONCLUSIONS

This study provides a nanoscale characterization of atmospheric aerosol particles collected in the Pantanal during an intense dry season, combining STXM at the C K-edge with nano-XRF elemental mapping. The results reveal pronounced intra-particle chemical heterogeneity, with clear gradients in carbon functional groups from the particle edge toward the core, indicating spatially resolved aging processes within individual aggregates.

This work highlights the powerful complementarity between advanced synchrotron-based techniques and conventional field measurements for atmospheric aerosol characterization. By providing access to physicochemical information at unprecedented levels of chemical specificity and spectral resolution, these techniques revealed features that cannot be fully captured through traditional approaches alone. The results demonstrate that significant opportunities remain for advancing our understanding of aerosol properties and atmospheric processes, emphasizing the potential of synchrotron science to open new research directions and contribute to future high-impact studies in atmospheric and environmental sciences.

Less oxidized, aromatic-rich carbon is predominantly located at the particle periphery, while more oxidized species are enriched in the core, demonstrating that atmospheric processing leads to chemically distinct microenvironments within single particles. Complementary nano-XRF measurements show significant variability in elemental composition and spatial organization, with larger particles dominated by mineral dust and smaller particles enriched in biomass burning tracers such as K, S, and Cl. The presence of mixed particles further supports the role of atmospheric aging and source mixing during transport. Variations in spatial

correlations between elements such as Cl, S, and Ca highlight differences in internal organization across particles, ranging from relatively homogeneous structures to highly heterogeneous aggregates. Taken together, these results challenge the assumption of internal homogeneity commonly adopted in atmospheric models, emphasizing the need to account for intra-particle variability in aerosol–cloud–climate interactions.

From a methodological perspective, this work demonstrates the potential of combining synchrotron-based techniques to investigate aerosols at the single-particle level. Although STXM and nano-XRF are widely applied in other fields, their use in atmospheric sciences remains limited. Their integration provides complementary insights into carbon speciation and elemental composition, enabling a more comprehensive understanding of aerosol structure and evolution. Despite limitations related to sample size and experimental constraints, the consistency of the observed trends supports the robustness of the findings. Future work will focus on correlative approaches to directly link chemical speciation and elemental distribution, advancing the understanding of aerosol formation and aging processes.

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6. GENERAL CONCLUSION

This thesis presents a multiscale and integrated framework for the characterization of atmospheric aerosols in tropical environments, focusing on the Pantanal and Central Amazon biomes. By combining real time measurements with advanced synchrotron-based techniques and complementary spectroscopic methods. It was possible to access the physicochemical complexity of aerosols from the bulk scale down to the nanoscale at same time. We successfully achieved the integration of field measurements and laboratory analyses, providing a more comprehensive characterization of aerosol microphysical properties.

The results demonstrate a strong size-dependent variability in chemical composition, oxidation state, and mixing characteristics, reflecting distinct formation pathways and atmospheric aging processes. In addition, a significant influence of anthropogenic emissions was observed, especially during the dry season, with increased concentrations of fine particles, black carbon, and trace metals associated with biomass burning and dust resuspension. Long-range transport was also identified as a relevant driver of aerosol composition in the Amazon, reinforcing the dynamic and interconnected nature of the atmospheric system.

The integration of XRF, XPS, FTIR, NEXAFS, and STXM revealed a level of chemical and structural heterogeneity that cannot be captured by conventional bulk approaches, emphasizing the limitations of traditional methodologies. In this context, this work demonstrates that multitechnique and multiscale strategies are essential to advance the understanding of aerosol formation, transformation, and environmental impact. Additionally, the findings presented here contribute to reducing key uncertainties in the representation of aerosols in atmospheric and climate models, particularly regarding aerosol–cloud interactions and indirect radiative effects. This thesis highlights the importance of detailed investigations in underrepresented tropical regions and establishes a robust foundation for future studies integrating advanced characterization, atmospheric modeling, and long-term monitoring to better constrain the role of aerosols in the Earth system.

Finally, this work also represents a process of personal and scientific growth. The PhD should be seen not merely as the final stage of academic training, but as the first step toward intellectual autonomy and the ability to make decisions that can positively influence society. The path taken here reflects a commitment to science, to

the pursuit of knowledge, and, above all, to the responsibility of transforming knowledge into collective benefit.

Statement on the Use of Artificial Intelligence

During the preparation of this thesis, generative artificial intelligence tools were used exclusively for editorial assistance with English language revision, grammatical correction, text organization, and the development of computational codes for data processing and visualization. The scientific content, experimental design, analyses, interpretations, conclusions, and the original manuscript were conceived, developed, and written by the author. The AI tools were not used to generate scientific content, produce scientific results, interpret experimental data, or formulate the conclusions of this work. All content presented in this thesis is the sole responsibility of the author.

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