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Can tree-ring chemistry be used to monitor atmospheric nanoparticle contamination over time?

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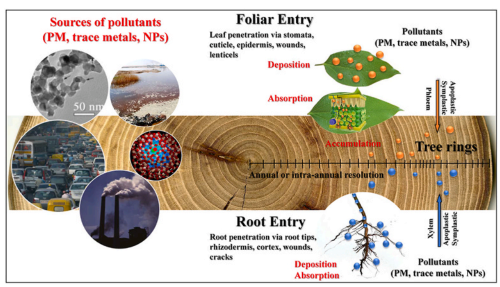
HIGHLIGHTS

- Trees can be used as bioindicators and proxy recorders of past air pollution events.
- Potential uptake and translocation of nanoparticles into the tree rings is possible.
- Dendrochemistry could be a promising tool to monitor nanoparticle contamination.

GRAPHICAL ABSTRACT



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ABSTRACT

Industrial activities and human population growth have resulted in an unprecedented increase in the release of particulate matter (PM) into the environment. Nanoparticle (NP) contamination is widespread and affects all terrestrial and aquatic ecosystems, putting humans and environment at risk. Several studies on the impact of PM and NPs on human health have been conducted over the past two decades, but their effects on plants are still poorly understood. What happens to them in forest ecosystems and trees has yet to be explored. In this paper, we review the literature on the capacity of trees to be used as bioindicators and proxy recorders of past air pollution events. Current research indicates that ultrafine particles can be taken up and translocated to different parts of a tree by physical and chemical processes, as we present studies of plant uptake and translocation processes of NPs in trees. Tree-ring chemistry, i.e., dendrochemistry, has been successfully used to reconstruct trace metal deposition from a variety of sources of pollution, including cars, metal refineries and coal burning. The use of dendrochemistry in environmental monitoring seems promising particularly given the presence of recent development of analytical tools, and is likely to provide useful data on atmospheric NP contamination that could not be obtained from any other source. However, still relatively little is known about the dynamic relationships between NPs and trees. We therefore discuss what future dendrochemical research is needed to make dendrochemical analyses as accurate as possible for monitoring atmospheric nanoparticle contamination over time.

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1. Introduction

1.1. Background

Air pollution is one of the most important problems related to industrialisation and human population growth. Its effects on the environment and on human health are a major concern. In Europe and in Northern America, many air pollution disasters stand out historically for their impact on human health, with consequent effects on public perceptions, scientific research and government regulation (Alfaro-Moreno et al., 2013). Following the Great Smog of 1952 in London (Bell et al., 2004) and the promulgation of the "Clean Air Act" in 1956 in the United Kingdom, air pollution became a topic of global concern.

Particulate matter (PM) components of air pollution have altered the chemical composition of the atmosphere, water and soil. Atmospheric PM, or aerosol particles, consist of complex mixtures of liquid and solid particles of organic and inorganic substances suspended in the air (WHO, 2016). The particles vary in size, sources, chemical composition and properties (see Fig. 1, Luo et al., 2019). Hazardous heavy metals, such as arsenic (As), chromium (Cr), lead (Pb), nickel (Ni), zinc (Zn), mercury (Hg), cadmium (Cd), and vanadium (V), may attach themselves to and co-exist with various aerosol particles (Shahid et al., 2017). The mixtures of air pollutants emitted by factories, households (mainly through cooking and heating), and vehicles are complex, and many are harmful to human health (WHO, 2018). High mortality rates are mainly due to exposure to small particles up to 250 nm in diameter ($PM_{0.25}$), called quasi-ultrafine fractions, that have more severe effects on human health than larger-size fractions that range from 2.5 μm ($PM_{2.5}$) to 10 μm (PM_{10}) (Lanzaco et al., 2019). Health effects of long-term exposure to low concentration of $PM_{2.5}$ have mostly been investigated on populations of high socio-economic status, but the risk of mortality seems to be higher among self-identified racial minorities and people with low incomes (Di et al., 2017).

After dry or wet deposition (Luo et al., 2019), acidifying pollutants and trace metals can be incorporated into soil and water or further transported *via* soil erosion, surface runoff, and groundwater flow (Wright et al., 2018). The effects of these pollutants on aquatic and terrestrial environments include degradation of marine ecosystems (Luoma, 1996; e.g., Waterhouse et al., 2012), wetlands and peatlands (Hutchinson and Meema, 2013), groundwater (Yazdi and Vosoogh, 2019), materials' corrosion (Vidal et al., 2019), soil acidification by sulphur and nitrogen deposition (Lamers et al., 2013; Huang et al., 2014a), with consequent damage to microbial communities (Wright et al., 2018), vegetation (Zhang et al., 2017) and wildlife (Sepp et al., 2019). Pollutant uptake and toxicity in biota can be affected by physical, chemical and biological factors (Wright et al., 2018). In plants, besides the well-known root uptake, the pathways of leaf deposition and foliar absorption were recently shown to contribute significantly to the pollutants' accumulation (Luo et al., 2019).

The ultrafine fraction of PM with at least one dimension less than 100 nm is defined as nanoparticles (NPs; Nel et al., 2006) and they account for approximately 3–8% of the total amount of PM smaller than 2.5 μm (Dietz and Herth, 2011). Engineered NPs or ultrafine particles produced through manufacturing (i.e., ultrafine TiO_2), and combustion processes (i.e., vehicle exhaust, energy production), so called incidental, can be inhaled and translocated to lungs and other distant organs, causing inflammatory reactions and oxidative stress (Madl and Pinkerton, 2009; Nemmar et al., 2013). Although the production of NPs from natural sources seems to be significantly the greatest (5 times more than that from engineered sources), the toxicity of natural NPs, potentially

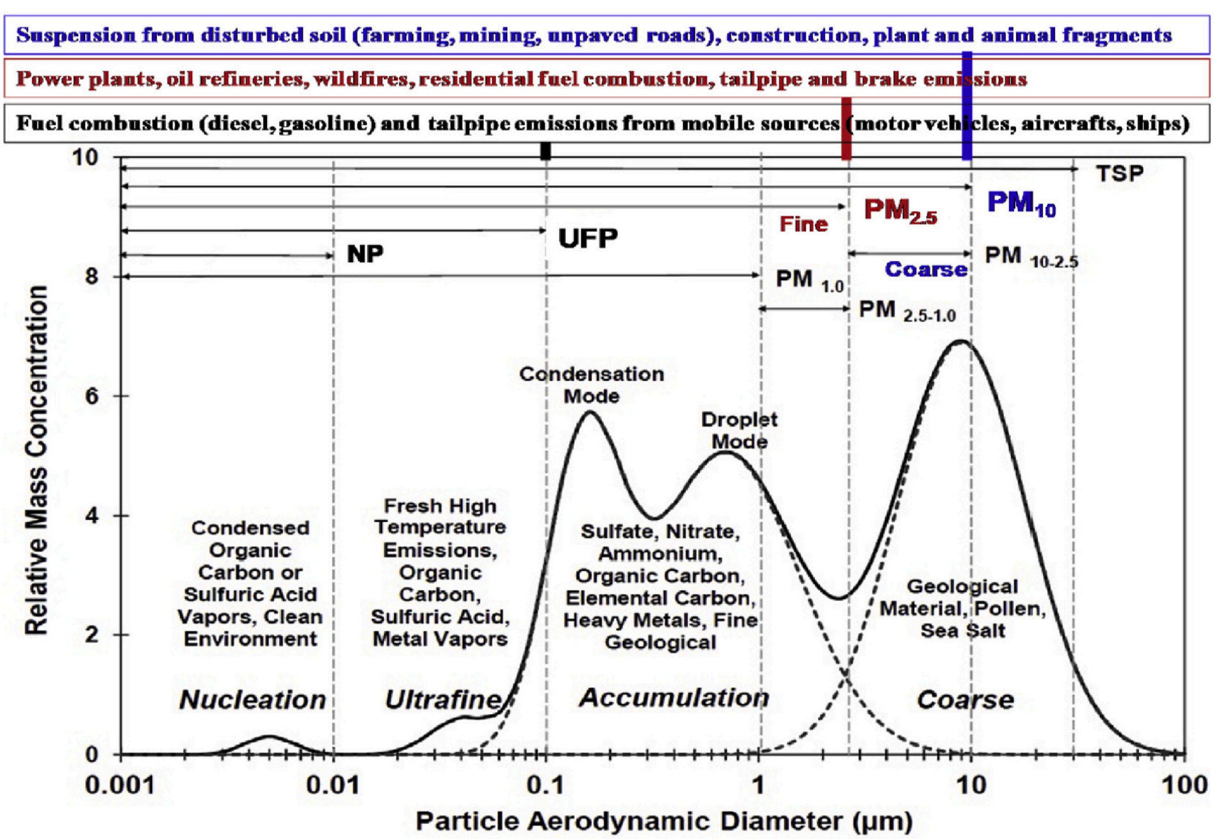

Fig. 1. Major sources of PM, size distribution and components. Anthropogenic particles are generated through combustion of fossil fuels, industrial activities, abrasion, and the re-suspension of natural particles by traffic, construction, and surrounding agricultural activities. Naturally occurring particles are produced by desert dust, sea spray, volcanoes, grassland fires, and a variety of biological sources. TSP: Total Suspended Particles; UFP: Ultrafine Particles; NP: Nanoparticles. (From Luo et al. (2019). Copyright 2019 by Elsevier Ltd).

vehicles for the transport of known toxic compounds, has received less attention than the potential toxicity of engineered NPs (Wigger et al., 2020). It has been reported that elevated concentrations of atmospheric nanocluster aerosol (i.e., particles with diameter range of 1.3–3 nm) mostly originates from traffic in urban areas (Rönkkö et al., 2017). They can be directly emitted from wheel brakes and be formed from initially gaseous exhaust compounds *via* nucleation in the atmosphere (Rönkkö and Timonen, 2019).

NPs released directly into water and soil or the atmosphere all end up in soil or water, either directly or indirectly, for instance, via sewage treatment plants, waste handling or aerial deposition (Nowack and Bucheli, 2007). High concentrations of NPs released in the environment (e.g., 7600 t/a of TiO_2 in sediments and 7000 t/a in landfills, 1300 t/a of ZnO NPs in sediments, 300 t/a in soil and 200 t/a in landfills) can have toxic effects on animals and other organisms (Bundschuh et al., 2018). Aggregated or adsorbed NPs in water or soil are less mobile, but uptake by sediment-dwelling animals or filter feeders is still possible (Nowack and Bucheli, 2007). In terrestrial ecosystems, engineered NP accumulation in plants is favoured due to exposure concentrations in the μg-mg kg^{-1} range (Schwab et al., 2016). Bioavailability of NPs also depends on specific properties of each NP, in addition to composition of the soil matrix, porosity, hydraulic conductivity, groundwater gradient and flow velocity, and geochemical properties (Remédios et al., 2012). Interactions between NPs and plants affect transformation and environmental fate of NPs. Plants can either have negative implications for human health as pollutant carriers into the human diet or positive implications due to atmospheric clearance (Dietz and Herth, 2011).

Measurement of atmospheric PM started before the seventies, but only during the seventies and eighties did it become possible to quantify the aerodynamic sizes of the particles and, therefore, identify the sources of different particles (Alfaro-Moreno et al., 2013). Identifying the source and characteristics of the PM is important for the development of pollution control strategies in order to evaluate human exposure to pollutants and assess their health-related risks (Huang et al., 2014b). Similarly, the occurrence and character of NPs in the environment are crucial for understanding the toxicology and ecological effects of increasing NP emission. Material flow analysis (production, manufacturing, use, disposal and recycling) and environmental fate models of engineered NPs could predict their environmental concentration (Wigger et al., 2020). As suggested by Tarrahi et al. (2020), plant nanotoxicology should also be taken into consideration for current risk assessments associated with NPs, their use, distribution and release. Moreover, determining the leaf uptake of NPs in trees of different heights could contribute to the investigation of the effect of vertical dispersion of NP concentration near a pollution source, such as a road, which are only available at < 5 m and 30 m above ground (Belkacem et al., 2021). Therefore, NPs concentration in other environmental ecosystems, like forest ecosystems, should be evaluated.

Real-time measurements of PM have several weaknesses due to technical, physical and economic limitations (Belkacem et al., 2021). Short-term air quality data from monitoring networks are widespread in urban areas in Europe and North America, but scarce in Africa, Asia, Oceania and South America (Lanzaco et al., 2019). Moreover, sampling of ultrafine particles is essential, notably in health-related studies, although standard protocols with longer-term objectives need to be set (Belkacem et al., 2021). Thus, to complement direct air pollution measurements and expand the still short period covered by instrumental records, biomonitoring methods can be considered (Muñoz et al., 2019). Terrestrial biomonitors of trace metals include, among others, mycorrhizal fungi, lichens and mosses (Shahid et al., 2017), bryophytes, dandelions and seagrass leaves (Wright et al., 2018). Trees also can uptake toxic elements and store them in woody tissues. A tree records a year-by-year signal during the entire period of its life, reflecting not only the tree's age, but also the environmental conditions under which the tree has been growing, including both the climatic conditions and air quality.

1.2. Rationale

In the current literature, only few studies have investigated the foliar penetration of airborne NPs in annual plants that are easy and fast to grow (e.g., Hong et al., 2014; Larue et al., 2014; Kranjc et al., 2018). Very little research has been done on the fate of nano-scale particles in trees, as well as on their penetration into the leaves. We report that trees take up nanoparticles through the leaves and transport them into the stem faster than through the roots. Recent results showed that foliar uptake seems to be the most efficient way for NPs and NP-associated pollutants' uptake and transport in trees (Cocozza et al., 2019), although the pathways used to translocate pollutants into the stem are still not clear. Through the detection of chemical elements in the tree rings, i.e., dendrochemistry, it is possible to reconstruct historical spatio-temporal pollution events (Binda et al., 2021), although the reliability of dendrochemistry has been questioned for some time (Watmough, 1997). New and innovative analytical techniques have the potential to investigate the presence of NPs in the tree rings and therefore allow the use of dendrochemistry in providing past levels and events of atmospheric NP contamination.

The aim of this review is therefore: a) to provide a synthesis of the impact of atmospheric PM on trees and their use as natural bioconcentrators of air pollutants, b) to discuss the current knowledge about the presence of ultrafine particles, i.e., nanoparticles, in trees, as well as their uptake and transport (with focus on the leaf uptake), and c) to explore the potentials and limitations of dendrochemistry as a tool to monitor atmospheric NP contamination over time. Finally, knowledge gaps that have been addressed by this analysis of the literature are summarized, and important areas for future research are identified.

2. Air pollution and trees

2.1. The impact of air pollution on trees

The impact of air pollution on vegetation downwind of point or urban polluting sources has been the subject of many studies (see, for a review, Innes, 1993; Innes and Haron, 2000; Innes and Oleksyn, 2000; Percy and Ferretti, 2004). Pollutant deposition on vegetation has been observed and analysed since the 1930s (Beath et al., 1935; Whitby, 1939; Sisam and Whyte, 1944). During the 1970s and 1980s, the health and vitality of forest ecosystems received much more public and political attention in connection with the decline of the forest observed in Central Europe. This decline is still believed to be connected to air pollution and acidic deposition (Schütt and Cowling, 1985), although recently it was proposed to have been induced also by drought (Cherubini et al., 2021). The first concerns about air pollutants were over carbon dioxide (CO_2), sulphur dioxide (SO_2), nitrogen oxides (NO_x) and ammonia (NH_3). Evident effects of NO_x and SO_2 deposition, mainly from power plants and industry, were well documented in Germany, Great Britain and other Central European countries, as were negative effects on tree growth and extensive forest dieback (Ulrich and Pankrath, 1983). Notable damage to forests due to SO_2, fluorides, and oxidants has also been reported in the United States and Canada (Miller and McBride, 1975). However, it is worth reporting that air pollutants, namely PM, NPs or dust deposition, can also cause beneficial effects on forest ecosystems by suppling essential nutrients (e.g., nitrogen, phosphorus) and microbial communities for vegetation growth (e.g., Magnani et al., 2007; Yu et al., 2015; Kameswaran et al., 2019).

Crown foliage transparency, defoliation and tree-ring width have been commonly believed to be the best indicators of tree condition in relation to air pollution. However, tree-ring stable isotopes have been used to study past forest decline episodes, and may integrate these indicators and enhance our understanding of tree vitality (Cherubini et al., 2021). Although the subject of considerable controversy over the years (Skelly and Innes, 1994), forest condition is influenced by air pollution. Higher plants have been used for many years as bioindicators of toxicity and as natural bioconcentrators of air pollutants.

2.2. The impact of trees on air pollution

The beneficial effects that trees have on air quality have been broadly documented in the literature since at least the 1930s (Beckett et al., 2000; Leung et al., 2011; Xie et al., 2011). Nevertheless, in some cases trees are known to negatively affect air quality and consequently human health, e.g., through the release of wind-dispersed pollen and of biogenic volatile organic compounds (Grote et al., 2016; Samson et al., 2017; Eisenman et al., 2019). Trees have been reported to be capable of effectively filtering pollutants out of the air, intercepting them with their bark, branches and leaves, where they then accumulate (Turan et al., 2011; Nowak et al., 2013; Han et al., 2020). In their review, Barwise and Kumar (2020) comprehensively examined the aspects of plants that influence the air quality, underlining the importance of species-specific traits, such as leaf characteristics and species suitability. Trees are thought to remove atmospheric PM through particle adsorption on their leaves and trace metals primarily *via* uptake by leaf stomata, cuticular cracks, lenticels, ectodesmata and aqueous pores (Hu et al., 2014; Shahid et al., 2017). Although the possibility of canopy uptake of pollutants has been less considered, results from Lockwood et al. (2008) showed that atmospheric organic nitrates can be taken up by foliage and incorporated into the leaf amino acids. The authors suggested that the uptake occurred mainly through the stomata rather than through the cuticle, which agrees with the results of Gessler et al. (2002). Elements absorbed by leaves could be partly translocated to roots or leached from plant foliage, especially by acid rain (Sensuła et al., 2021). Analysing tree components has been widely used as a low-cost alternative method to determine the spatial distribution of trace metals and estimate current atmospheric levels. For example, the levels of PM originating from the nearby incinerator and other pollution sources in the Venafro Plain (Molise, Italy) were recorded in the wood, bark and leaves of downy oak (Cocozza et al., 2016). Pb isotopic composition of pine needles indicated that Pb isotope ratios are also a good indicator to evaluate the degree of air pollution in heavily industrialized areas (Sensuła et al., 2021). Besides the spatial distribution of trace elements, trees are indeed able to provide their temporal distribution by finding associations between industrial history and the pattern of chemical elements in the tree rings, as recently demonstrated by Cocozza et al. (2021).

2.3. Tree rings as archives of atmospheric particulate matter

Since the beginning of the 1970s, dendrochemistry is described as a related research area of dendrochronology, i.e., the study of tree growth over time (Lepp, 1975). Dendrochemistry is used to analyse trace element concentrations in annual rings and to determine historical changes in trace metal levels in soils, sediments and the atmosphere. Tree components such as leaves or bark can be used to determine the spatial variations of current atmospheric pollution in a region, but only tree rings can be used to assess both spatial and historical variations of past pollution trends and events (Odabasi et al., 2016).

The ability of trees to accumulate contaminants from their surroundings has been demonstrated in a number of studies, and trace metal levels in wood appear to correlate with changes in metal emissions from suspected pollution sources (e.g., Barnes et al., 1976; Ferretti et al., 1993; Padilla et al., 2002; Richer-Laflèche et al., 2008). Trace element concentration in tree rings can show the efficiency of decontamination policies for reducing the air pollution in a certain region, as well as the timing of the reduction of the pollutant concentration in the ecosystems surrounding the polluted area (Liu et al., 2018; Locosselli et al., 2018; Muñoz et al., 2019).

Trees take up inorganic and non-volatile contaminants and their non-volatile tracers or biodegradation by-products, which are often transported as part of soluble organic compounds (ligands), and become fixed as part of the tree's wood tissues (Balouet et al., 2015). The distribution of trace metals in tree rings is mainly thought to depend on xylem transport, which represents water and contaminants taken up by the roots from the soil or ground water (Balouet et al., 2015). However, the level of contamination and other soil properties, e.g., pH, redox potential, texture, and organic matter content, can limit the transport of pollutants (Burken et al., 2011). Trees cannot always access contaminated groundwater, and soils with high organic content or clay lenses make root uptake difficult, as Rocha et al. (2019) observed.

The pathway of leaf translocation seems to contribute more efficiently to the trace element accumulation in tree rings under specific conditions, and especially with atmospheric pollution (Luo et al., 2019). Arnold et al. (2018) found that *Populus tremuloides* could take up atmospheric Hg and that *Pinus nigra* could translocate highest Hg concentration from foliage to tree rings through stomatal and nonstomatal uptake mechanisms. Foliar uptake of NO_x from vehicle exhausts near a motorway in Switzerland has been detected in the tree rings of Norway spruce through nitrogen stable isotope analysis; $\delta^{15}N$ was found to increase after the construction of the motorway, and was also lowest in trees further away from the source of pollution, suggesting increased foliar uptake of NO_x from traffic exhaust rather than from nitrogen deposition on soil (Saurer et al., 2004).

Recently, some studies have confirmed that nanomaterial can also accumulate in woody plants mainly through the leaf uptake (see *3.2 Uptake and transport of nanoparticles*) and be transported to the stem (see *3.3 Nanoparticles and trees*), although the uptake and presence of NPs in trees are still largely unexplored.

3. Nanoparticles and plants

3.1. The impact of nanoparticles on plants

In the last decade, concerns over the potential impact of NPs in the environment on aquatic and terrestrial organisms have increased (Baun et al., 2008; Handy et al., 2008; Navarro et al., 2008). Nanotechnology has been increasingly applied in a broad range of sectors, due to the unique general properties and the high diversity of elemental and structural composition of the NPs (Jun-Nam and Lead, 2008). Recent research trends suggest that nanotechnology will play an increasingly important role in agriculture (Su et al., 2019). Given that the literature is rich with studies about interactions of engineered NPs with organisms and in ecosystems, we will mainly refer to these when describing the potential toxicity, uptake and transport of NPs in plants. However, as Wigger et al. (2020) suggested, it is crucial to evaluate all nanoscale materials (engineered/incidental/natural NPs, dissolved metals, even micro- and nanoplastics) and related dynamic process in the same context.

Potential applications of nanotechnology have also been discussed for forest production systems, forest protection (Marimuthu et al., 2017) and forest management (Singh et al., 2021). NPs have mostly been used in the controlled release of agrochemicals such as biostimulators in fertilizers to increase plant growth (e.g., Aleksandrowicz-Trzcinska et al., 2018) and disease resistance (Liu and Lal, 2015; Adhikari et al., 2016), in pesticides and herbicides to improve pest and disease management (Wang et al., 2016), and in target-specific delivery of biomolecules (e.g., nucleotides, proteins and activators) (Torney et al., 2007; Martin-Ortigosa et al., 2014). Engineered NPs, such as nano-TiO_2, CeO_2 and ZnO, have also been used to control toxic metals in soil through phytoremediation techniques (Singh and Lee, 2016), and silver (Ag) NPs to control fungicides (Kim et al., 2009; Nhien et al., 2018). However, it is assumed that only 0.1% of applied crop-protection-agents reach their biological target, whereas the majority are lost to the environment (Karny et al., 2018). Thus, this innovative technology poses concerns about their environmental discharge and the risk of potentially adverse effects on human health and natural systems (Anjum et al., 2016; Bundschuh et al., 2018).

Increasing concerns have considered absorption, translocation, bioavailability and toxicity of NPs in plants, although some impacts of NPs on plants' physiological and biochemical parameters have been positive (see review of Liu et al., 2020). Generally, direct toxic effects have been observed in many annual species, and even in trees (Seeger et al., 2008; Sweet and Singleton, 2015). Depending on their concentrations and characteristics, NPs have been found to damage the physiology and membrane patterns in herbaceous species (Shaw et al., 2014). Engineered NPs mainly seem to negatively affect plants due to their chemical composition and surface reactivity (Rastogi et al., 2017). Ag and TiO_2 NPs can induce oxidative stress, cytotoxicity and genotoxicity, thus affecting plants' germination, growth and photosynthesis (Cox et al., 2016). As shown by Izad et al. (2019), high concentrations of iron oxide NPs may affect the leaf gas exchange and the biochemical processes (chlorophyll content and stomata density) in oil palm seedlings. The effects of NPs may also be indirect, leading to physical restraints, the release of toxic ions or the production of reactive oxygen species (ROS) (Bundschuh et al., 2018). Nevertheless, whether the effects are inhibiting, promoting or even lacking may vary according to the plant species and the characteristics of the NPs (Du et al., 2017).

3.2. Uptake and transport of nanoparticles

NPs and dissolved ions from metallic engineered NPs in sewage sludge and nano-agrochemicals (Lv et al., 2019) can enter into the food chain. Direct uptake and accumulation in plant tissue can occur from contaminated soil/water or from the atmosphere (Ma and Yan, 2018). Two pathways for the delivery of NPs into the plant system after their deposition onto a plant surface have been well described in recent reviews: root-to-leaf and leaf-to-root (see Fig. 2; Schwab et al., 2016; Su et al., 2019). The upward transport system through the xylem differs markedly from the downward transport system through the phloem in both structure and function. Thus, the uptake, translocation and accumulation of NPs in plants depend on chemical composition, concentration, size and dimension, chemical and physical properties of plant species (Sanzari et al., 2019).

NPs are adsorbed onto plant surfaces and taken up through natural nano- or micrometer-scale plant openings due to the hygroscopic affinity between particles and the tissue surface of the leaves (Burkhardt et al., 2012). Although several studies confirmed the root uptake (mainly through wounds and cracks at lateral root junctions; James and Olivares, 1998) and the xylem transport of NPs in herbaceous species (Zhu et al., 2008; Cifuentes et al., 2010; Peng et al., 2015), the epidermis and the Casparian strip (in fully formed roots) may block the penetration of NPs (Su et al., 2019).

Compared to the root uptake of NPs, leaf uptake has been less studied. The transport path of NPs in the foliar uptake can be driven by two main processes: through the cuticle (about 0.6–4.8 nm, via diffusion and permeation or via polar aqueous pores) and through the stomatal openings (Eichert et al., 2008). The stomatal pathway is the only confirmed pathway for the foliar uptake of NPs from the leaf surface to the internal tissues (Lv et al., 2019). A recent study showed that NPs (95 ± 25 nm) could penetrate the leaf of tomatoes and translocate in a bidirectional manner, distributing to other leaves and to the roots (Karny et al., 2018). Leaf surface area and evapotranspiration may be good estimators for assessing to what extent NPs could be taken up into the plant via the stomata (Navarro et al., 2008). Investigations on NP leaf uptake have been mainly related to agricultural practices (e.g., targeted delivery, fertilizers, pesticides) on edible plants, such as pumpkin (Corredor et al., 2009), watermelon (Wang et al., 2013), lettuce (Zhao et al., 2016), soybean and tomato (Li et al., 2018). What happens to the NPs in trees is still under debate.

3.3. Nanoparticles and trees

Concerns related to NPs as atmospheric pollutants mainly deriving from traffic (Kangasniemi et al., 2019; Manigrasso et al., 2019) and the combustion process (Tissari et al., 2019) started to receive scientific attention only in the last decade. NPs deposited over aerial surfaces can, eventually, be transported via phloem (after entering the leaf apoplast through the stomatal pathway) or assimilated by a tree's roots if deposited on soil and transported via xylem to the cambium zone at different tree heights (see Fig. 3; Ma et al., 2017; Arnold et al., 2018). Although there are some studies about the effects of NPs used in forestry applications as growth stimulators, fungicides, to improve pest resistance on trees (e.g., Bao-Shan et al., 2004; Kim et al., 2009; Olchowik et al., 2017; Aleksandrowics-Trzcinska et al., 2018; Su et al., 2020),

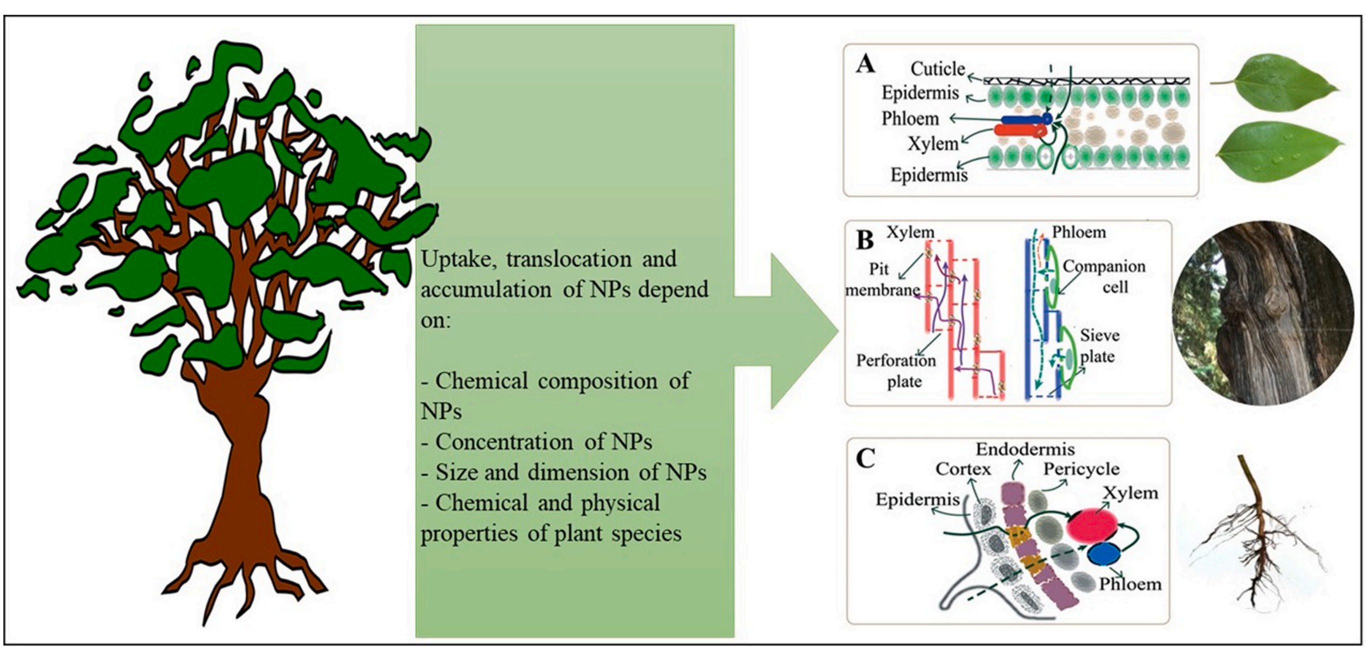

Fig. 2. Key micro-morphological and physiological indices of plants affecting the fate and transport of NPs in plants are represented. Cross/longitudinal section of A) leaf, B) xylem and phloem in a stem, and C) root. Arrows show possible entry pathways and delivery of NPs within plant tissues (Adapted from Su et al. (2019). Copyright 2019 by The Royal Society of Chemistry).

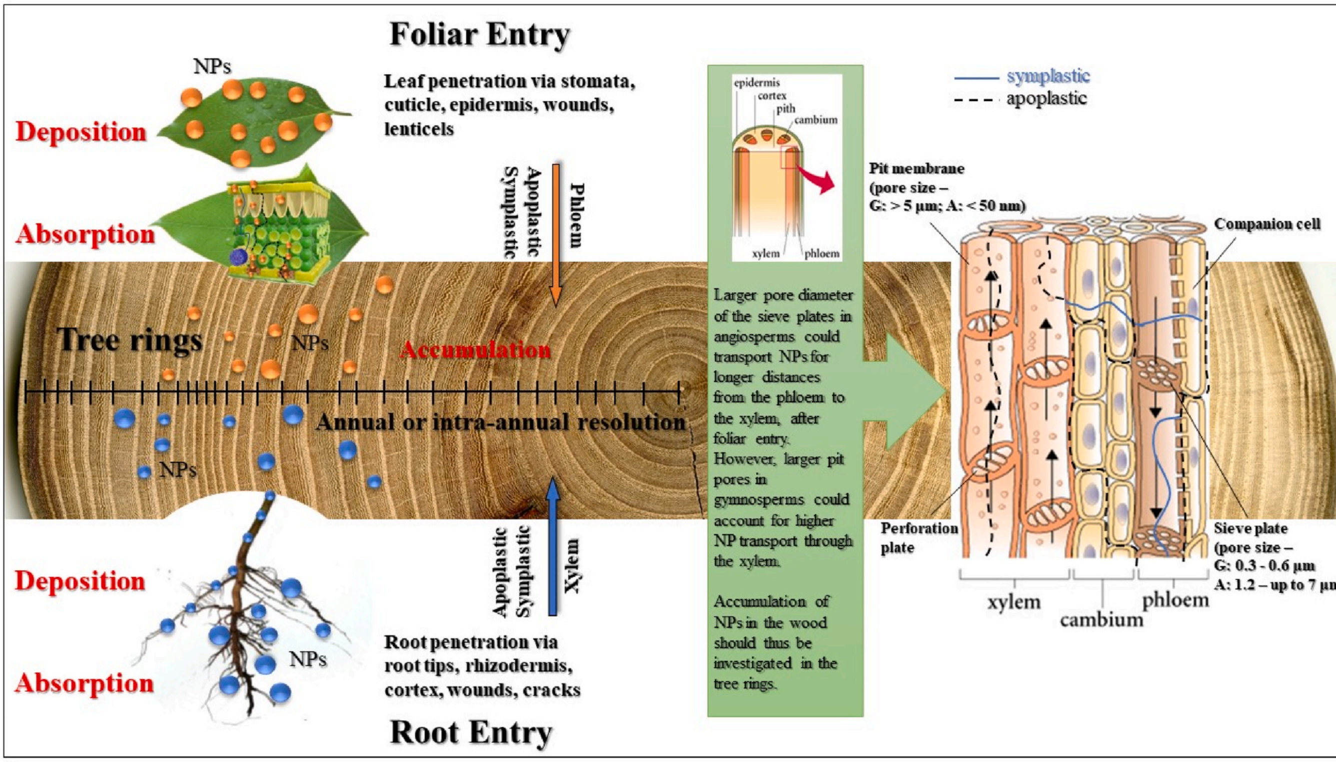

Fig. 3. Schematic of potential nanoparticle accumulation in the tree rings after foliar and root entry in trees. After their uptake, NPs can be transported through the xylem (root entry) or through the phloem (foliar entry) to the tree rings via symplastic and/or apoplastic pathways. The transport efficiency of the NPs strongly depends on the tree species and pore sizes of the conductive system. NPs: nanoparticles; A: angiosperms; G: gymnosperms (anatomical illustration licensed under CC BY-SA-NC).

there is little knowledge about their uptake and transport in these woody perennial plants.

Fluorescence microscopy revealed aqueous apoplastic movement through the cell wall and into the phloem after foliar penetration of NPs smaller than 5.4 nm in citrus leaves (Etxeberria et al., 2016). Water-suspended hydrophilic particles of bigger sizes (43 nm or 1.1 µm) have been shown to penetrate through the stomata of *Vicia faba* and *Allium porrum* (Eichert et al., 2008). Similarly, inert TiO_2 fine particles (270 nm) have been found to enter the intercellular space of birch leaves *via* stomata (Räsänen et al., 2017). Nevertheless, it is not yet clear how these NPs cross the plant membrane (whether through endocytosis or channels – aquaporins and plasmodesmata), when they move using symplastic pathways or how they cross the endodermis/Casparian strip (Tripathi et al., 2017).

The NP (as dissolved complex or particulate material) translocation and accumulation in trees was investigated by Cocozza et al. (2019). The authors found a greater amount of Ag in the stem of foliar treatment than of root treatment of oak, Scots pine and poplar, suggesting the phloem transport of NPs from leaves to the stem along with photosynthates. The findings of Su et al. (2020) suggested that the mechanism of delivery is driven by steric repulsive interactions between the NPs and the conducting tube surfaces of phloem and xylem, during petiole feeding and truck injection of Ag NPs in citrus trees.

The phloem (symplastic) and the xylem (apoplastic) are complex conductive systems that are essential for the long-distance transport of NPs in trees (Fig. 3). Sap composition of xylem and phloem can affect the aggregation, sedimentation and dissolution of NPs and, thus, could modify the size exclusion limits and surface properties, whereas the sap flow rate can determine the transport velocity of NPs (Su et al., 2019). In particular, meta-analysis of experimental data about phloem transport in trees have shown that the average transport speed is two times faster for angiosperm trees than for gymnosperm trees (Liesche et al., 2015), which could suggest greater transport of NPs in angiosperms. The higher hydraulic resistance in gymnosperm trees can be directly related to their phloem anatomy which is characterized by longer sieve elements and smaller sieve pores (ranging from 0.3 to 0.6 µm) than in angiosperm trees (pores ranging from 1.2 to 7 µm; Liesche et al., 2015). The pit membranes of the xylem conductive cells are designed to restrict the passage of air bubbles and pathogens. Although different techniques used for sample preparation can explain large variation in pore sizes (up to 700 nm for dried samples), fresh samples showed sizes below 50 nm in various angiosperm species (Zhang et al., 2020). Thus, some NPs might accumulate in the tracheids and vessel elements of the xylem having their transport restricted by the pit membranes (Corredor et al., 2009). Electron microscopy showed that pit pore traits are also highly variable in gymnosperm trees. The dimensions of the pit pores are on the order of microns in diameter and vary in different parts of the same tree. Schulte et al. (2015) observed larger pit apertures in roots (5.3 and 5.7 µm in *Picea mariana* and *Picea glauca*, respectively) than in stems (3.3 and 3.2 µm). The mean diameter of bordered pits may vary from higher in earlywood fibers to lower in latewood fibers and with the age of cambium (Sirviö and Kärenlampi, 1998). Thus, large pit apertures and high pit membrane porosity (Schwab et al., 2016) could account for highest NP transport through the xylem in gymnosperm trees. Moreover, low NP translocation in drought-tolerant plant species (i.e., high segmentation of the xylem) with low water transpiration rates, and vice versa, showed that NP uptake rates correlate with the transpiration in vascular plants (Schwab et al., 2016). Uptake rates for insoluble NPs in poplar, and into shoots, were reported to be 3.8, 3.7 and 0.83 µg kg^{-1} d^{-1} for 15, 25 and 50 nm gold NPs, respectively (Zhai et al., 2014), several orders of magnitude lower than for soluble NPs.

The detection and characterization of NPs in above ground tissues demonstrates the ability of trees to take up and translocate NPs, although the exact pathways and mechanisms of uptake and transport of

NPs are still unclear. Laser Ablation Inductively Coupled Plasma Mass Spectrometer (LA-ICP-MS) has proved to be a valuable analytical tool for bioimaging of metal species in plant leaves (Ko et al., 2019) and roots (Wojcieszek et al., 2019), as well as in tree rings (e.g., Sensuła et al., 2017; Perone et al., 2018; see paragraph *4.4 Analytical techniques*). Even though LA-ICP-MS is not able to provide information about the physico-chemical form of the metal analysed, it can be successfully applied when the information about the presence of the metal in its nanoparticulate form has been confirmed by another technique (Wojcieszek et al., 2020), such as imaging techniques (Yan and Chen, 2018) and single particle ICP-MS (Peters et al., 2015).

4. Recommendations for reliable dendrochemical analyses

4.1. Potentials and limitations of dendrochemistry

Tree-ring growth is influenced by such a large variety of factors that determining the effect of a single chemical element or compound is difficult (Ferretti et al., 2002). Tree-ring growth is a very unspecific measure, and the same effect may have many different causes. How suitable dendrochemistry is for providing unbiased evidence of the impacts of trace metal pollution has been controversial (Szopa et al., 1973; Hagemayer et al., 1992). Different environmental and growing conditions could have an influence of the uptake and concentration of trace metals in the tree rings (Liu et al., 2009). Thus, the date of the tree ring in which an element is detected may not be the year in which the element was released into the environment, described as the “hysteresis” phenomenon (Lukaszewski et al., 1988; Bellis et al., 2004; Liu et al., 2018). Nevertheless, the major limitation of dendrochemistry appears to be the radial translocation of chemical elements. Trace metals may be translocated across the full width of the sapwood, which constitutes a major problem in dendrochemistry (Watmough, 1997; Scharnweber et al., 2016). The chemical elements that are transported in the xylem sap inside tree vessels can migrate between tree rings through radial translocation (Lepp, 1975), which interferes with how the environmental pollution is recorded in the tree rings (Smith and Shortle, 1996). Radial translocation occurs along ray cells and can be linked to internal biological processes such as sapwood senescence, heartwood formation or fungal infection (Meerts, 2002), as well as wood characteristics of tree species (see paragraph *4.2 Tree selection*). The process of heartwood formation can be associated with parenchyma cell death, disappearance of storage material and increase in extractive content (Piqueras et al., 2020). Stewart (1966) suggested that the formation of the first cylinder of heartwood could also occur when waste metabolites, i.e., extraneous materials as well as trace metals and PM, that are transported along the xylem rays and accumulated in the region of the pith, reach a toxic level. However, some observations showed that heartwood does not function primarily for waste storage (Taylor et al., 2002). The death of parenchyma cells will cause the sapwood-heartwood boundary to move outwards as the diameter of the tree increases (Stewart, 1966). Only sapwood rings translocate waste elements, and if the sapwood is formed by a limited number of rings, the date of tree rings in which an element is detected is not with annual-resolution but with a longer-term resolution. For example, if the number of rings in the sapwood is ten, the resolution will be a decade.

Wood parenchyma rays can transport trace metals from the outer rings to the inside of the sapwood. Essential elements, such as Cu, P and Zn, tend to increase towards the outer rings to meet the metabolic needs of the cambium (Speer, 2010), whereas non-essential elements, such as Cd and Pb, show no radial tendency (Watmough, 1999). Liu et al. (2018) found that P and Zn concentrations increased in the outermost rings, probably as a result of inter-ring migration. In contrast, Zhang (2019) detected translocation of Zn and Pb within the tree rings, but no translocation of Cu.

Other limitations of dendrochemistry may reasonably be dependent on tree selection, sampling methods and analytical techniques, which will be discussed in the next paragraphs. Nevertheless, a very detailed methodological guide provided by Balouet et al. (2015) gives information on best practices about sampling methods, sample preparation, analytical methods and data handling when using the dendrochemical approach to document pollution history recorded in trees.

4.2. Tree selection

Not all trees are suitable for dendrochemical studies (Lepp, 1975). Cutter and Guyette stressed in their review (1993) that anatomical, chemical and ecological factors should be considered in choosing which tree species to use. Of these, the main criteria for the selection of tree species should be the environmental conditions in which they grow, the xylem traits and the trace metals under study. Conifers are, according to Legge et al. (1984), more suitable than broadleaf species for detecting heavy-metal elements because they have tracheids and fewer short ray cells. Among the broadleaved species, Lepp (1975) suggested that ring-porous species, such as *Quercus, Ulmus, Fraxinus*, are better suited for dendrochemical analysis as they possess long xylem elements with large lumina. They therefore cavitate very soon after the ring is formed, almost every winter, isolating the chemical signal stored in the ring. However, it should be noted that large parenchyma rays, such as those in *Quercus* sp., which are absent in conifers, may allow radial translocation of chemical elements (see paragraph *4.1 Potentials and limitations of dendrochemistry*).

The selection of trees must also consider the absorptive capacity of the chemical elements, which means that some trees are able to absorb particular elements better than others. For example, Liu et al. (2018) found that the contents of Cd and Zn in white poplar were higher than in *Ailanthus* sp., which indicates the absorptive capacity of white poplar is better for Cd and Zn.

The condition of the tree is also relevant. External signs on the trunk, such as injury, cracks in the pith and stem, or slight discoloration of the extracted tree core due to fungal infection are all indications that the tree is unsuitable for dendrochemical analyses (Smith and Shortle, 1996). In this context, it is worth mentioning that the practice of increment bore coring itself is in general considered not harmful for health of taxa from temperate ecosystems (Tsen et al., 2016). The effects of increment borer coring on the survival, health and growth of tropical species were highlighted in a review by Neo et al. (2017). The authors observed that increment coring does not negatively impact the survival and growth of some tropical species within the first year after coring, but it can provoke external damages, such as discoloured bark, surface wounds and presence of fungi or insects in the boreholes, often observed also in temperate coniferous and deciduous tree species. Moreover, particular care must be taken when selecting trees from tropical and sub-tropical regions, where precipitation is the main limiting factor for the tree growth and thus for the formation of annual rings (Cherubini et al., 2003).

Young trees are not suitable because they do not provide long-term tree-ring records. However, adult trees growing close to urban or industrial areas are crucial for dendrochemical studies but they might be rare. Thus, environmental protection agencies should identify and preserve them as part of a forest archive of air quality records, complementing conventional environmental monitoring at local or national scales (Alterio et al., 2020).

It should be noted that trees that are suitable for tracking trace elements might not be suitable for monitoring NP concentrations in the environment. However, we lack current knowledge about this point.

4.3. Sampling methods

Care must be taken with sample preparation to avoid contaminating the specimens with the elements being investigated (Smith and Shortle, 1996). For example, contamination might occur from dropping specimens on the ground, from not cleaning increment borers properly or from using contaminated reagents in the chemical procedures during sample digestion. An increment borer, the main field tool for collecting dendrochronological samples, can leave traces of iron, chromium and tungsten, which are its primary metal constituents, on the sampling cores. To cope with this problem, laser trimming can be used. It is a form of trimming that removes the outer surface of a core, resulting in a surface that is, according to Sheppard and Witten (2005), free of contamination. Instead of sanding the cores as usually done in dendrochronology or leaving them unprocessed, the best practice for sample preparation for dendrochemical analysis is cutting the surface using a microtome, as explained by Danek et al. (2015).

Interpreting dendrochemical data poses several problems, but careful sampling strategies and sample preparation should prevent and reduce the risk of contaminating the samples.

4.4. Analytical techniques

There is no single analytical method to identify or quantify all types of pollutants (inorganics or organics) (Balouet et al., 2015). The techniques most commonly used in dendrochemistry include Inductively Coupled Plasma Mass Spectrometer (ICP-MS), Proton Induced Gamma Ray Emission (PIGE), Neutron Activation Analysis (NAA), Ion Chromatography, X-ray fluorescence (XRF), and Differential Pulse Anodic Stripping Voltammetry (DPASV) (Watmough, 1999). In the past, most techniques were destructive, which involved the cutting, milling and digests of the wood, and were not able to perform multi-element analyses. Other chemical analysis techniques and instruments are far more precise (low detection limits) and enable the analysis of intact cores with annual, or even intra-annual, resolution if needed, i.e., Secondary Ion Mass Spectrometry (SIMS), Proton Induced X-ray Emission (PIXE) and LA-ICP-MS (e.g., Legge et al., 1987; Brabander et al., 1999; Loader et al., 2017). LA-ICP-MS is the only technique available for direct investigation of trace metals in tree rings (Binda et al., 2021), which could also be applied in combination to other techniques for NP identification, as previously mentioned. To correct for differences in ablation, ^{13}C is used as an internal standard. In order to avoid misinterpretation of trace element concentrations due to airborne dust or adhering soil particles on plant tissues, correction methods may need to be applied to obtain the "true" element concentration in plants (Pospiech et al., 2017).

Finally, besides all the above-mentioned precautions and according to the objectives of the research, to further improve the dendrochemical analyses, analytical techniques with low detection limits and high number of trace elements and isotopes analysed should be chosen.

5. Conclusions

The importance of trees as bioindicators of environmental pollutants, and thus their role in contributing to the atmospheric PM monitoring, as well as in association with areas of instrumentation designs and toxicology, is underestimated. During the past decades, many scientific studies have been investigating the capture of atmospheric PM by trees, with special attention paid to its foliar deposition. Recent research has shown that the foliar uptake of airborne particles, such as NPs, is possible, and that they may be translocated into different parts of a tree, including the annual rings. Dendrochemical studies have been widely used to monitor environmental changes in soil and atmosphere, successfully reconstructing trace metal deposition in tree rings since the early seventies.

The initial investigation discussed in Section *4 Recommendations for reliable dendrochemical analyses*, in relation to the previous Sections of this review, may be used in order to strengthen the reliability of dendrochemical methods when investigating the atmospheric PM and NP contamination over time.

Significant considerations and knowledge gaps include:

- Still relatively little is known about the dynamic relationships between NPs and trees. We must have some investigation of the main pathways NPs use to enter and leave the trees, as well as how these pathways differ in different tree species, in order to discover which parts of the tree will accumulate the most NPs, and which tree species are the most exposed and the most suitable for taking up NPs.
- When investigating NPs in the environment (and plants), it is important to consider that all types of NPs (natural, incidental, engineered) should be evaluated into an interconnected context (Wigger et al., 2020) that includes all dynamic processes that transform carbon, dissolved metals and particles into each other (e.g., NPs can form from dissolved metals). Moreover, nanoparticle traits (shape, size, surface charge, surface composition) have to be considered when studying their uptake and translocation in trees, and plants in general.
- If dendrochemical analyses are to be used to assess past atmospheric particulate matter, we will need to find out: i) which chemical elements are taken up by which tree species, ii) which physiological processes are involved in tree uptake in the different pathways (foliar, bark and root), and iii) where and how the different chemical elements are stored within the tree. In this regard, problems related to the suitability of selected trees and the radial translocation of some chemical elements could be overcome by drawing on a better understanding of tree physiology, wood characteristics and metal behaviour in wood.
- When interpreting tree-ring chemical composition, it should be noted that finding no evidence of a particular metal in the tree rings does not mean the element is necessarily absent.
- The study of the spatial distribution of metal-containing NPs in plant tissues could help to understand the exact pathways and mechanisms of uptake and transport of NPs, which are still unclear. Using a combination of multiple techniques could help in filling this gap.
- The isotope ratios of hydrogen, oxygen and carbon have been widely used in tree ring studies (McCarrol and Loader, 2004). Regarding other macronutrients, such as isotopic ratio of nitrogen, and minor and trace metals, such as Cu, Pb and strontium, have also been measured to determine their sources (Binda et al., 2021). Thus, the analysis of metal isotopes in tree rings could be considered for NPs detection as many metal-based NPs (that could form from dissolved metals) have multiple stable isotopes that can be precisely measured by LA-ICP-MS.
- Exploring the presence of nanoparticles in trees, could not only allow assessment of their spatial and temporal distribution, but could also help to asses NP concentrations in the environment.

This review provides a general overview of the capacity of trees to be used as proxy recorders of past air pollution events by absorption of pollutants in plant tissues and translocation into the tree rings. Moreover, this review reveals that tree-ring analysis has been applied to explore the impact of atmospheric pollutant concentrations on the chemical composition of tree rings.

In conclusion, research has shown that dendrochemical methods could provide a promising approach in future for monitoring atmospheric PM, including NPs. Moreover, future perspectives showed that sophisticated analytical techniques and the spectrum of elements analysed could increase, as well as time and costs taken to prepare samples with low risk of contamination could be reduced. Advancements towards such new and innovative analytical methods have been also presented by Binda et al. (2021). Further research, however, is needed (as outlined above) to understand the uptake and transport mechanisms of NPs in trees and to make dendrochemical analyses as accurate as possible.

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Declaration of competing interest

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