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From problems to solutions

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An overview of the key topics related to the study of tire particles and their chemical leachates: From problems to solutions

Farhan R. Khan [a,*], Elisabeth S. Rødland [b], Pieter Jan Kole [c], Frank G.A.J. Van Belleghem [c,d], Adrián Jaén-Gil [e], Steffen Foss Hansen [f], Alessio Gomiero [e]

[a] NORCE Norwegian Research Centre, Climate & Environment Division, Bergen, Norway
[b] Norwegian Institute for Water Research, Økernveien 94, 0579, Oslo, Norway
[c] Department of Environmental Sciences, Faculty of Science, Open Universiteit, 6419, AT Heerlen, the Netherlands
[d] Zoology: Biodiversity and Toxicology, Centre for Environmental Sciences, Hasselt University, BE, 3590, Diepenbeek, Belgium
[e] NORCE Norwegian Research Centre, Climate & Environment Division, Mekjarvik 12, 4072, Randaberg, Norway
[f] Department of Environmental and Resource Engineering, Technical University of Denmark, 2800, Kgs. Lyngby, Denmark

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ABSTRACT

The environmental contamination and biological impacts of tire particles and their associated chemicals is of great concern. Variously termed as tire wear particles (TWP), tire and road wear particles (TRWP), and end-of-life tires (ELTs) to differentiate between possible compositions and sources in the environment, these rubber particles are a major source of plastic pollution. The array of added chemicals that leach into the environment presents an additional environmental, toxicological and regulatory challenge. In this review, we define several topic areas that are relevant to the study of tire particles, outlining the current state and ongoing challenges. Emissions estimates needed to establish environmental loads are based on dated, summarized datasets rather than measured data, but determining the environmental presence of tire particles is hampered by a lack of standardised sampling methods. Several techniques for the analysis of tire rubber and associated chemicals are available, but the variability in tires model composition makes the selection of analytical markers challenging, as does a lack of commercial standards. Toxicologically, there remains a need to delineate particle and leachate effects and, in addition, to investigate emerging chemicals of concern and their transformation products. Such knowledge is required to feed into regulatory processes which currently regulate tire constituents separately rather than as the whole tire. Technological solutions to capture or replace tire rubber are progressing, often outside of academia, and it is important that the scientific community collaborate with those attempting to reduce the environmental impact of tires, including the tire industry.

1. Introduction

The modern tire is a highly complex feat of engineering comprised mainly of blends of styrene-butadiene rubber, polybutadiene rubber and natural rubber (i.e. polymer/elastomer 40–50 %) and strengthened and compounded with inorganic fillers (e.g. carbon black, silica 30–35 %) [1,2]. To this are added a range of chemicals in the form of emollients (hydrocarbons, ~15 %), additives (antioxidants, plasticizers, 5–10 %) and vulcanization agents (2–5 %). Formulations vary between manufacturers and models in efforts to optimise tires that perform according to the desired properties of wear resistance, rolling resistance and wet traction in different driving conditions (e.g. summer vs. winter tires) [3]. Within the emollients, additives and vulcanization agents are numerous chemicals that fall within the groups of organics (e.g. benzothiazoles, phthalates and polycyclic aromatic hydrocarbons, 6PPD) [4,5] and trace metals (predominantly zinc, but also copper, lead and cadmium [6,7]).

Variously termed as tire wear particles (TWP), tire and road wear particles (TRWP), and end-of-life tires (ELTs) to differentiate between possible compositions and sources in the environment, these rubber particles have been described as a major contributor to plastic pollution in the world's aquatic habitats [8]. The contamination of the environment and biological impacts of tire particles and their associated chemicals is of great concern both within and in addition to the current focus on microplastic pollution [9]. The leaching of the aforementioned

* Corresponding author.
E-mail addresses: fakh@norceresearch.no, farhan.khan@gmx.com (F.R. Khan).

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chemicals further adds to the complexity of their environmental fate and potential risks. Of the >200 organic substances detected in tires, almost 60 % were leachable, with the intrinsic polarity of the chemical being a strong determinant of its aquatic mobility [10]. Tire particles and their leachates have been shown to cause toxicity and adverse outcomes to a variety of aquatic organisms, with some specific chemicals and/or chemical degradation products now being pinpointed as known toxic agents, for instance 6PPD and 6PPD-quinone [11,12].

The complexity of the research area presents itself in several ways, from the heterogeneity of tire formulations that vary across manufacturers and models, to the distinct forms being researched as an environmental pollutant (i.e. tire and road wear particles (TRWP)) and tested within the laboratory (i.e tire wear particles (TWP)), to the mixture of chemicals that are added to and leach from the tire particles. These uncertainties, amongst others, present an on-going challenge to researchers attempting to detect and analyse, understand the toxicology and adverse effects of, and then regulate and mitigate, tire particles and their leachates. Whilst there are now several reviews on different aspects of tire research, such as occurrence and generation, analytical methodologies, and toxic effects [1,9,13–16], other topics such as emissions [17] and regulation [18] are less well-described in the literature. In this review paper, we define several topic areas that are relevant to the study of tire particles, outlining the development and current state of each topic, as well as highlighting the ongoing challenges within the field.

Nomenclature

Similar to the 'Are we speaking the same language?' paper by Hartmann and colleagues calling for a unified terminology for microplastics research [19], tire/tyre particle research has suffered from the application of inconsistent nomenclature [9]. 'Tire tread abrasion', 'tire debris', 'tire rubber', 'worn tread rubber', 'tire wear particles', 'roadway particles', 'tire components', 'tread particles', 'tire and road wear particle' and 'road dust' [6,9,16,20,21] have all been used to describe 'non-exhaust, traffic-related particulate matter' [1]. In attempting to apply consistency, Halle et al. (2020) [9] proposed the following clarification: (1) tire-road-wear particles (TRWP) as being abraded from tires when used on the road and this form is the most likely to be sampled from the environment; (2) tire-wear particles (TWP) that do not include the road component and the likely form generated for laboratory experiments; and (3) end-of-life-tires (ELT) i.e. repurposed tires used in, for example, artificial turf. Umbrella terms such as 'microrubber' [9] or 'tire microplastics' [15] have also been suggested for collectively describing tire-related particles or linking tire particles to microplastics but have not been extensively adopted. The term 'tire particle' has gained some common usage [22].

2. Emissions

Estimating the amount of tire particles released into the environment is complicated by numerous variables [23]. Vehicle weight has a proportional relationship with tire wear and tear. Greater wear is caused by increased acceleration and speed, braking harder, insufficient tire pressure and hotter conditions, whilst driving in wet conditions causes less wear [24]. However, driving behaviour is considered to be the biggest factor in the rate at which tires wear [24]. Based on the challenges of producing a global emission estimate, two approaches have been developed to obtain country-level emissions. The first uses an emission factor per vehicle kilometre multiplied by the distance driven. Emission factors are available as universal figures that can be combined with national mileage figures. Emission factors can be measured in real world driving by measuring the weight loss of the tires over a given distance. The second approach uses the average weight difference between an abandoned tire and its new counterpart and multiplies this figure by the number of abandoned tires in a country [25]. India has the lowest wear and tear, estimated using the "emission factor x distance driven" method, of 0.23 kg/capita/year, while the United States has the highest at 4.7 kg/capita/year, calculated from the average of values produced "emission factor x distance driven" and "tire weight difference" methods.

Whilst such studies have become established in the tire literature, a recent analysis of country-based emission estimates reveals reliance on summarized, rather than measured, data [17]. Most of these summaries originated in the 1970s and are limited in scope. Tire properties have evolved over time, becoming more durable, cars are heavier and engines more powerful, all of which influence wear rates. For example, Dannis (1974) [26] estimated a wear rate of 360 mg/km/car based on measurements of weight loss, but the German Automobile Club (ADAC), currently the only organization publishing figures on tire wear, measured 120 mg/km/car [27]. If both values are correct, tire wear has decreased by a factor of three in 50 years. Again, there is a discrepancy between the two methods of emission estimates, where studies using the average weight loss of abandoned tires estimated an average loss of 2.0 kg/year/capita, whereas studies employing emission factors per km reported 1.3 kg/year/capita [17].

To know the scale of tire wear entering the environment, there is an urgent need to obtain reliable emission data. It is vital to reduce uncertainties and find agreement between the different methods of calculation. The United Nations Economic Commission for Europe (UNECE) Working Party on Noise and Tyres (GRBP) established the Task Force on Tyre Abrasion (TFTA). Their objective is to advise on different methods for determining tire weight loss [28]. Even in the absence of reliable estimates, the issue of tire emissions gains traction, as exemplified by the current EURO7 proposal, which aims to reduce pollutant emissions from vehicles and includes limits on tire wear [29].

4. Analysis and detection

4.1. Analysis of the tire particle

Two main strategies are used to identify and quantify tire particles in environmental samples, either as single particles or as mass quantities (Fig. 1). The possibilities and limitations of these methods are discussed in detail elsewhere [3]. Research questions, availability of instrumentation, and costs are drivers behind the choice of methods in most studies since standardized analytical guidelines for tire particle analysis remain absent.

Single particle methods can provide outputs such as the number of particles, size, morphology, surface texture and color. Single particle methods that have been used to identify tire particles, but more specifically TRWP, are light microscopy (LM) [30,31], various versions of Fourier-transform infrared spectroscopy (μ-FTIR, ATR, μ-ATR-FTIR, STA-FTIR-PARAFAC) [32], Raman spectroscopy [33], scanning electron microscopy combined with energy dispersive x-ray spectroscopy (SEM-EDX) [34]. Using LM, particle shape, color and surface texture are used to identify potential TRWP in a sample. However, confirmation of TRWP is difficult without additional chemical markers for identification. Using μFTIR/(μ)-ATR-FTIR and Raman, spectra of the rubber component in TRWP may be possible to obtain; however, the high carbon black content in modern tires influences the quality of the spectra, making it difficult to determine if the particle is a TRWP or a different rubber particle containing carbon black. Using single-particle methods, the mass of TRWP in the sample may be calculated using the number of particles, size and density. However, assumptions about the three-dimensional form of small-sized particles are needed, as the single-particle methods only measure size in two dimensions. TRWP are also reported to have variable density, ranging from 1.3 to 2.1 g/cm^3, depending on the chemical content of the tire tread and the degree of mixture with minerals and other road particles.

Mass-based methods focus on identifying the presence of TRWP based on chemical markers and quantifying the mass of TRWP based on the level of these markers in the sample based on standard chemical

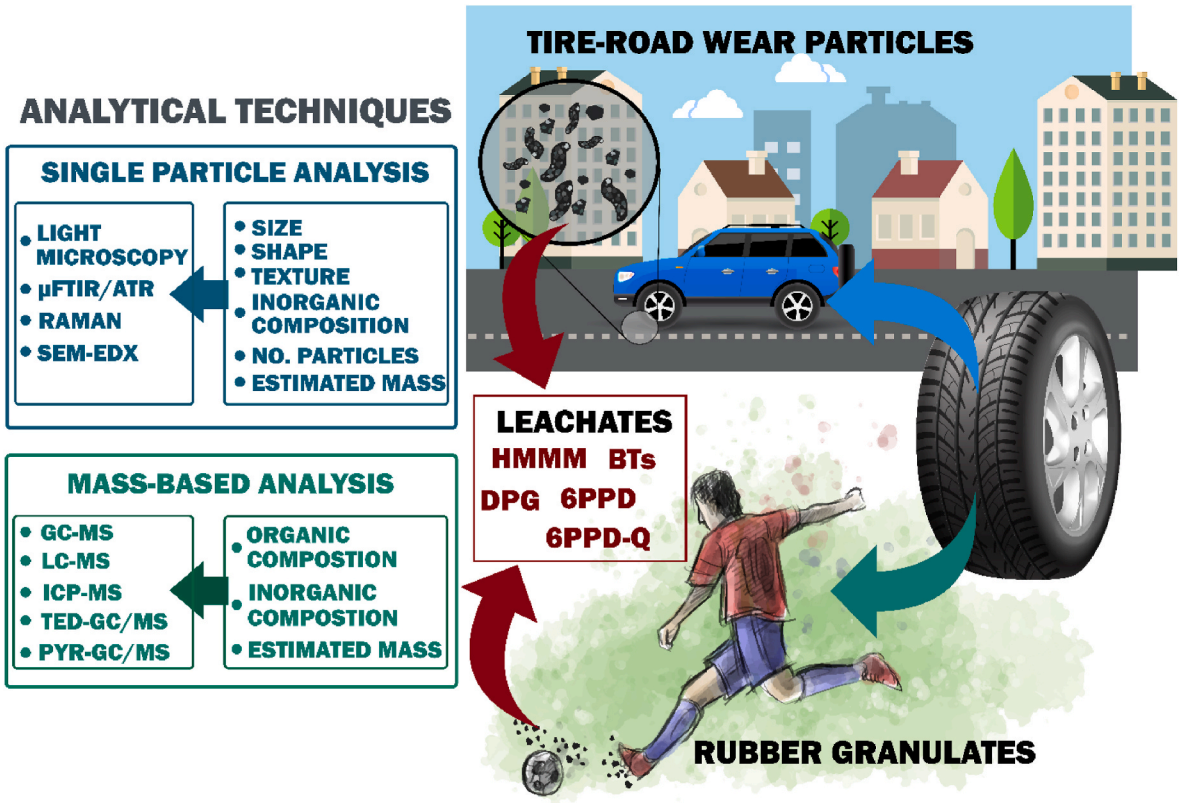

Fig. 1. The analysis of tire particles and their leachates is conducted using multiple analytical techniques based on the particle or sample mass. Illustration made with vectors from Freepik and Macrovector (www.freepick.org).

references (ex SBR1500, [35]), local reference tires [36] or global averages in tire tread [37]. Methods used for TRWP analysis include inductively coupled plasma mass spectrometry (ICP-MS), liquid chromatography mass spectrometry (LC-MS), gas chromatography mass spectrometry (GC-MS), pyrolysis and thermal desorption coupled to gas chromatography mass spectrometry (PYR-GC/MS, TED-GC(MS). One of the main challenges using mass-based methods is the selectivity and stability of markers. The characteristics of suitable markers include that they are not easily leached from the tire, that they are sufficiently specific to tires and not equally present in, for example, brake wear, and that they are analytically accessible [38]. Commonly used markers for the different mass-based methods are summarized in Table 1.

The variability of different chemical compounds between commercial tires [3] complicates the calculations from marker concentration to TRWP concentration further. Moreover, the aging of tire particles in the environment may change the chemical and physical characteristics of tire particles compared to new tire tread particles [7], which again potentially impacts the markers used for mass-based analysis [52]. Some studies use values from locally relevant reference tires to calculate the TRWP concentrations, whereas other studies apply global average shares across different brands and types of tires [38]. As TRWP concentrations are calculated differently, comparisons between studies are challenging. A global open-sourced tire database has been suggested as an option for researchers and public authorities to use the same values for specific areas, seasons, or vehicles [53].

As TRWP are heterogeneous particles, with a range of characteristics from the original tire tread as well as variation in their impact from environmental factors, complementary methods are needed to study the complexity of these particles in the environment. Recent studies have investigated tire tread material from a road simulator, TRWP from sediment and stormwater runoff with µFTIR/FTIR-ATR, SEM/SEM-EDX and PYR-GC/MS [54].

4.2. Analysis of the leachates

Gas and liquid chromatography coupled to tandem mass spectrometry have been widely applied for the detection of organic chemicals in complex aqueous matrices, providing high sensitivity for quantification in monitoring studies. Among the conventional tandem mass spectrometers, triple quadrupole and quadrupole-linear ion traps have exhibit excellent performance for the detection and quantification of *target* compounds commonly using multiple reaction monitoring (MRM) methodologies [55]. However, the low mass resolution attained prevents the elucidation of the *untargeted* compounds when reference standards are not available *a priori* [56]. Increases in tire performance in terms of rolling resistance, wear resistance and wet traction have resulted in significant differences between the formulations of different tire models from different manufacturers [57]. The variability in chemical composition creates new analytical challenges since the presence of tire leachates should be explored individually. However, most studies have only focused on the analysis and quantification of *targeted* pollutants leaching from tire materials (e.g. benzothiazoles, phthalate esters, and polycyclic aromatic hydrocarbons [58–61] with less attention paid to the high number of *untargeted* chemical compounds at lower concentration levels [62–64]. Overlooking their presence reduces the ability to understand the leachate composition and thus hampers the association between measured toxicity and chemical occurrence [63].

The use of advanced analytical instrumentation based on high-resolution mass spectrometry (HRMS), such as Fourier transform ion cyclotron resonance (FT-ICR), time-of-flight (TOF) and Orbitrap, has allowed the simultaneous detection of thousands of chemical components in a single sample analysis with confident assignment of elemental composition for enhanced identification [65]. Data acquisition modes, including data-dependent acquisition (DDA), data-independent acquisition (DIA), and data-independent all ion fragmentation (AIF) are used for method development, where both full scan HRMS and tandem mass

Table 1
Summary of different markers used to quantify TRWP in environmental samples the last 10 years. Note for Pyr-GC/MS, there were no new methods or marker changes between the publications of 2013 and 2021, as studies performing Pyr-GC/MS applied the methods by Unice et al., 2013 [35] in that time. SBR= Styrene-Butadiene Rubber, BR = polyButadiene Rubber, NR=Natural Rubber.

Analytical technique	Marker compound	Reference
Headspace gas chromatography mass spectrometry (HS-GC-MS)	Benzene, benzoic acid, phenol	Thomas et al., 2022 [39]
GC-MS	Oleamide	Chae et al., 2021 [40]
Liquid-chromatography tandem mass spectrometry (LC-MS/MS)	BT, 2-OHBT,2-Mercaptobenzothiazole (2-MBT), 2-Methylthiobenzothiazole (2-MTBT), 2-Aminobenzothiazole (2-ABT)	Bye and Johnson, 2019 [41]
Liquid-chromatography high resolution mass spectrometry (LC-HRMS)	N-Formyl-N-1,3-dimethylbutyl-N-phenylendiamine (N-Formyl-6-PPD), 1,3-dimethylbutyl-N-phenyl-endiamine-quinone (6-PPDQ), hydroxylated N-1,3-dimethylbutyl-N-phenyl quinone diamine (QDI-OH)	Klöckner et al., 2021 [5]
Inductively coupled plasma mass spectrometry (ICP-MS)	As, Bi, Cd, Co, Cr, Cu, Ni, Pb, Sb, S, Zn	Klöckner et al., 2019 [42]
	Zn	Klöckner et al., 2021 [43]
Thermal desorption gas chromatography mass spectrometry (TED-GC/MS)	4-Vinylcyclohexene (SBR + BR), Cyclo-hexenyl benzene (SBR) Isoprene (NR)	Eisentraut et al., 2018 [44]
	3-phenylcyclohexene (SBR)	Klöckner et al., 2019 [43]
	Cyclo-hexenyl benzene (SBR)	Müller et al., 2022 [45]
Pyrolysis gas chromatography mass spectrometry (Pyr-GC/MS)	4-Vinylcyclohexene (SBR + BR), Isoprene (NR)	Unice et al., 2013 [35]
	4-Vinylcyclohexene (SBR + BR), Isoprene (NR)	Panko et al., 2013 [46]
	4-Vinylcyclohexene SBB, SB (SBR + BR)	Goßmann et al., 2021 [36]
	4-Vinylcyclohexene, SB dimer, SBB trimer (SBR + BR)	Rauert et al., 2021 [47]
	Benzothiazole (tyre additives)	Parker-Jurd et al., 2021 [48]
	Butadiene dimer (SBR + BR) and isoprene dimer (NR)	Youn et al., 2021 [49]
	Benzene, α-methylstyrene, ethylstyrene, butadiene trimer A, styrene (SBR + BR)	Rødland et al., 2022 [50]
	Styrene (SBR + BR), Dipentene (NR)	Sun et al., 2022 [51]

spectra (MS/MS) are recorded without prior knowledge of the compounds present [55,66]. Such analytical strategies have been used with other pollutant groups, e.g. pharmaceuticals, organophosphate flame retardants, pesticides and personal care products [64,67,68], but studies with tire leachates remain scarce. The main drawback lies in the instrumental and data processing complexity as well as in the lack of commercial reference standards to finally confirm their presence [3]. Among the data reported, Müller et al. (2022) [10] characterized the leachables generated from nine tires in artificial freshwater within the experimental period of 28 days. The results showed the detection of 214 organic substances, of which 145 were classified as leachable and 13 were potential transformation products. Seiwert et al. (2020) [69] identified a high percentage of positive features (up to 85 % in urban creeks and 67 % in municipal wastewater) from a list of 48 suspected compounds that may be related to TRWPs. However, contributions from other sources could not be discounted. Similarly, Chibwe et al. (2021) [4] concluded that the presence of several identified benzothiazoles and arylamines correlated with the toxic effects measured in the embryos. Although new studies on the presence of suspected and unknown chemicals from tires constantly emerge, efforts to develop fast and reliable suspect and non-target screening methodologies are still needed. A comprehensive characterisation of a tire's environmental chemical fingerprint underpins the understanding of environmental presence and ecotoxicological effects, as discussed in the next sections.

5. Environmental presence

The occurrence of TRWP has been identified in a variety of environmental compartments, including air, road and tunnel road dust, road runoff, tunnel wash water, roadside snow, roadside soil, freshwater sediments, river water, wastewater treatment plants (WWTP), gully-pots, stormwater, and tunnel treatment sediments and in bioretention cells [1,14,38,50] (Table 2). Particles smaller than 10 μm (in the Particulate Matter range of <10 and < 2.5 μm i.e., PM10 and PM2.5) are likely to be removed from the road surface by wind and turbulence (air) and potentially deposited into terrestrial or aquatic environment. Levels of TRWP in the PM2.5 range between 0.012 and 6.5 μg/m3, PM10 range between 0.2 and 1.25 μg/m3. Fig. 2 and Table 1 summarize the current available data on TRWP mass concentration in the environment based on peer-reviewed studies.

Particles >10 μm are potentially deposited on the road surface and from there, the pathways depend on local conditions [1]. In dry weather, TRWP are deposited on the road surface (road dust 0.4–70 mg/g; tunnel road dust 7.7–204 mg/g) (Table 2). During precipitation, the TRWP are potentially transported into the environment through road runoff (1.5–40 mg/L) and often reach a downstream water body (river water: 0.6–179 mg/L; river sediment: 0.036–155 mg/g; lake sediment: 0.73 mg/g). Runoff and spray of TRWP from the road can also reach nearby soils (soil 0–1 m: 4.9–20.2 mg/g; soil >1 m; 0.7–13.4 mg/g; soil >20 m: 0.12–0.9 mg/g). In colder conditions, TRWP can accumulate in roadside snow. Some highways and high-traffic roads often have drainage systems that often contain high levels of TRWP (gully pots 0.2–130 mg/g, sedimentation basins 0.4–130 mg/g and bioretention cells 19.8–30.9 mg/g).

The currently available data on TRWP show a wide range of concentrations across the different environmental compartments and sample types, as well as a large range of concentrations within environmental compartments. This is potentially explained by variation in traffic density, traffic speed, driving behaviour, weather conditions, or other related variables impacting the release and transport of TRWP, as have been explored for road-side snow and road-side soils [94]. However, the currently available data within different environmental compartments is limited, and there is a need to include a variation in types of roads in future studies to better understand how different traffic variables and other factors influence the levels of TRWP in the environment. Another explanation for the observed variations in environmental concentrations is the lack of standardization of sampling methods, and in the pre-treatment and analysis of TRWP for different sample types. The currently available literature, lacks method validation and cross-method validation, making comparison between studies and critical assessment of TRWP levels in different compartments challenging [3]. Assessing whether inter-study variability of TRWP occurrence and concentrations is due to methodological differences or actual environmental differences is a vital research need.

The currently available data represents only a few countries, and it is important to understand how differences in transport patterns affect the environmental presence of TRWP. Germany (n = 8), Norway (n = 5), Japan (n = 4) and China (n = 3) have the highest number of studies on TRWP mass concentrations (Table 2). Two studies were found for the USA and the UK and only one study for Australia, France, Italy, Korea, Malaysia, Sweden and Switzerland. Given the geographic limitations and studies per matrix, it remains difficult to draw general conclusions

Table 2
Mass concentration levels of TRWP in different environmental compartments (air deposition, natural environment, road environment and treatment) compiled from current available peer-reviewed literature. Single values are average values reported, and for studies not reporting average values, the range (minimum-maximum) were applied instead. Different methods and different markers for the different methods have been applied.

Matrix	Type	Concentration	Unit	Analysis (marker)[a]	Country	Reference
Ambient air	*Air deposition*	2008	μg/m^3	AAS (Zn)	Denmark	(Fauser et al., 1999) [20]
Air PM10		300	μg/m^3	Pyr-GC/MS-FPD (TR)	Japan	(Panko et al., 2019) [70]
Air PM10		1250	μg/m^3	Pyr-GC/MS-FPD (TR)	United Kingdom	(Panko et al., 2019) [70]
Air PM10		200–500	μg/m^3	ICP-MS (Zn)	Germany	(Fomba et al., 2018) [71]
Air PM80-1		1400–2400	μg/m^3	SEM-EDX automated	Sweden	(Järlskog et al., 2022) [72]
Air PM80-1		1510–13500	μg/m^3	SEM-EDX automated	Switzerland	(Rausch et al., 2022) [73]
Air PM2.5		22	μg/m^3	Pyr-GC/MS-FPD (TR)	Japan	(Panko et al., 2019) [70]
Air PM2.5		42	μg/m^3	Pyr-GC/MS-FPD (TR)	United Kingdom	(Panko et al., 2019) [70]
Air PM2.5		12	μg/m^3	Pyr-GC/MS-FPD (TR)	USA	(Panko et al., 2019) [70]
Air PM 2.5		6500	μg/m^3	Pyr-GC/MS (TR)	China	(Liu et al., 2023) [74]
Lake sediment	*Natural environment*	0.73	mg/g	ICP-MS (Zn)	Germany	(Klöckner et al., 2019) [42]
River sediment		0.35–7.4	mg/g	Pyr-GC/MS (TR)	France	(Unice et al., 2013) [75]
River sediment		0.072	mg/g	Pyr-GC/MS (TR)	Germany	(Goßmann et al., 2021) [76]
River sediment		0.4–3.9	mg/g	GC/MS/FPD (NCBA, 24MoBT)	Japan	(Kumata et al., 2000)[b] [77]
River sediment		0.046–2.8	mg/g	Pyr-GC/MS (TR)	Japan	(Unice et al., 2013) [75]
River sediment		0.092–0.59	mg/g	GC/FPD (NCBA)	Malaysia	(Zakaria et al., 2002) [78]
River sediment		10–155	mg/g	GC/MS (24MoBT)	USA	(Spies et al., 1987)[a] [79]
River sediment		0.48–1.4	mg/g	Pyr-GC/MS (TR)	USA	(Unice et al., 2013) [75]
River water		0.8–18.5	mg/L	Pyr-GC/MS (TR)	Australia	(Rauert et al., 2022) [80]
River water		0.8	mg/L	GC/MS (24MoBT, BT)	China	(Ni et al., 2008) [81]
River water		0.6–179	mg/L	GC/MS/FPD (NCBA, 24MoBT)	Japan	(Kumata et al., 2000)[b] [77]
Roadside snow >60 m		37.9	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022b) [82]
Soil >1 m		0.71	mg/g	TED-GC/MS (TR)	Germany	(Müller et al., 2022) [45]
Soil >1 m		0.1–2.3	mg/g	Pyr-GC/MS (TR)	Japan	(Unice et al., 2013) [75]
Soil >1 m		3.7–4.8	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022c) [83]
Soil >1 m		13.5	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2023b) [82]
Soil >20 m		0.12	mg/g	TED-GC/MS (TR)	Germany	(Müller et al., 2022) [45]
Soil >20 m		0.56–0.9	mg/g	Pyr-GC/MS (TR)	Japan	(Unice et al., 2013) [75]
Soil 0–1 m		20.2	mg/g	ICP-MS (Zn)	Germany	(Kocher et al., 2008) [84]
Soil 0–1 m		8.5	mg/g	TED-GC/MS (TR)	Germany	(Müller et al., 2022) [45]
Soil 0–1 m		4.9	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022c) [83]
Soil 0–1 m		11.3	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2023b) [82]
Road dust	*Road environment*	0.47	mg/g	Pyr-GC/MS (TR)	China	(Sun et al., 2022) [51]
Road dust		35–70	mg/g	TED-GC/MS (TR)	Germany	(Eisentraut et al., 2018) [85]
Road dust		1.7–11.3	mg/g	Pyr-GC/MS (TR)	Germany	(Goßmann et al., 2021) [76]
Road dust		4.5–37	mg/g	GC/MS/FPD (NCBA, 24MoBT)	Japan	(Kumata et al., 2000)[b] [77]
Road dust		0.4–19	mg/g	GC/MS/FPD (NCBA, 24MoBT)	Japan	(Kumata et al., 2002)[b] [86]
Road dust		9.8–22.6	mg/g	Pyr-GC/MS (TR)	Korea	(Youn et al., 2021) [49]
Road dust		1.4–8.4	mg/g	GC/FPD (NCBA)	Malaysia	(Zakaria et al., 2002) [78]
Road dust		35	mg/g	GC/MS (BT2-OHBT)	USA	(Rogge et al., 1993) [87]
Road dust		3–43	g/m3	Automated SEM-EDX	Sweden	(Järlskog et al., 2022) [72]
Road dust Tunnel		110–120	mg/g	ICP-MS (Zn)	Germany	(Klöckner et al., 2021) [88]
Road dust Tunnel		7.7–204	mg/g	GC/MS/FPD (NCBA, 24MoBT)	Japan	(Kumata et al., 2000)[b] [77]
Road dust Tunnel		12–55.1	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022a) [89]
Road runoff		31–40	mg/L	Pyr-GC/MS (TR)	Italy	(Rosso et al., 2023) [32]
Road runoff		1.5–8.2	mg/L	Pyr-GC/MS (TR)	United Kingdom	(Parker-Jurd et al., 2021) [90]
Roadside snow 0–1 m		563	mg/L	GC/MS (BT, MeSBT, MeBT)	Germany	(Baumann and Ismeier, 1998)
Roadside snow 0–1 m		3122	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022b) [91]
Roadside snow 1–2 m		2137	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022b) [91]
Roadside snow 3–4 m		566	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022b) [91]
Tunnel wash water		33.6	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022a) [89]
Bioretention cell	*Treatment*	19.8–30.9	mg/g	STA-FTIR (Tire tread)	Norway	(Mengistu et al., 2022) [92]
Gully-pot		0.2–150	mg/g	STA-FTIR (Tire tread)	Norway	(Mengistu et al., 2021) [93]
Gully-pot		3.5	mg/g	Automated SEM-EDX	Sweden	(Järlskog et al., 2022) [72]
Gully-pot - Tunnel		6.87–76.8	mg/g	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022a) [89]
Sedimentation basin - sediment		0.4–130	mg/g	TED-GC/MS (TR)	Germany	(Eisentraut et al., 2018) [85]
Stormwater sediment		14.5	Mg/g	Automated SEM-EDX	Sweden	(Järlskog et al., 2022) [72]
WWTP effluent		0.02	mg/L	Pyr-GC/MS (TR)	United Kingdom	(Parker-Jurd et al., 2021) [90]
Tunnel wash water (treated)		12.5	mg/L	Pyr-GC/MS (TR)	Norway	(Rødland et al., 2022a) [89]

[a] Methods: Gas chromatography mass spectrometry (GC/MS), Pyrolysis-GC/MS (Pyr-GC/MS), Pyr-GC/MS-flame photometric detection (Pyr-GC/MS-FPD), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Thermal Desorption-GC/MS (TED-GC/MS), Simultaneous Thermal Analysis-Fourier-transform infrared spectroscopy (STA-FTIR). Markers: Various pyrolysis markers for styrene butadiene rubber (SBR), butadiene rubber (BR) and natural rubber (BR), summarized here as markers for tire rubber composition (TR), zinc (ZN), Benzothiazole (BT), N-cyclohexyl-2-benzothiazolamine (NCBA), 2-(4-Morpholinyl)benzothiazole (24MoBT), 2-hydroxy benzothiazole (2-OHBT), 2-methylthiobenzothiazole (MeSBT), 2- Methylbenzothiazole (MeBT), tire tread.
[b] TRWP levels calculated based on the reported tire chemical values.

about the presence and distribution of TRWP in the environment.

6. Toxicology and adverse effects

6.1. Bioavailability and toxicology

Early research into the potential toxicity of tires was conducted by

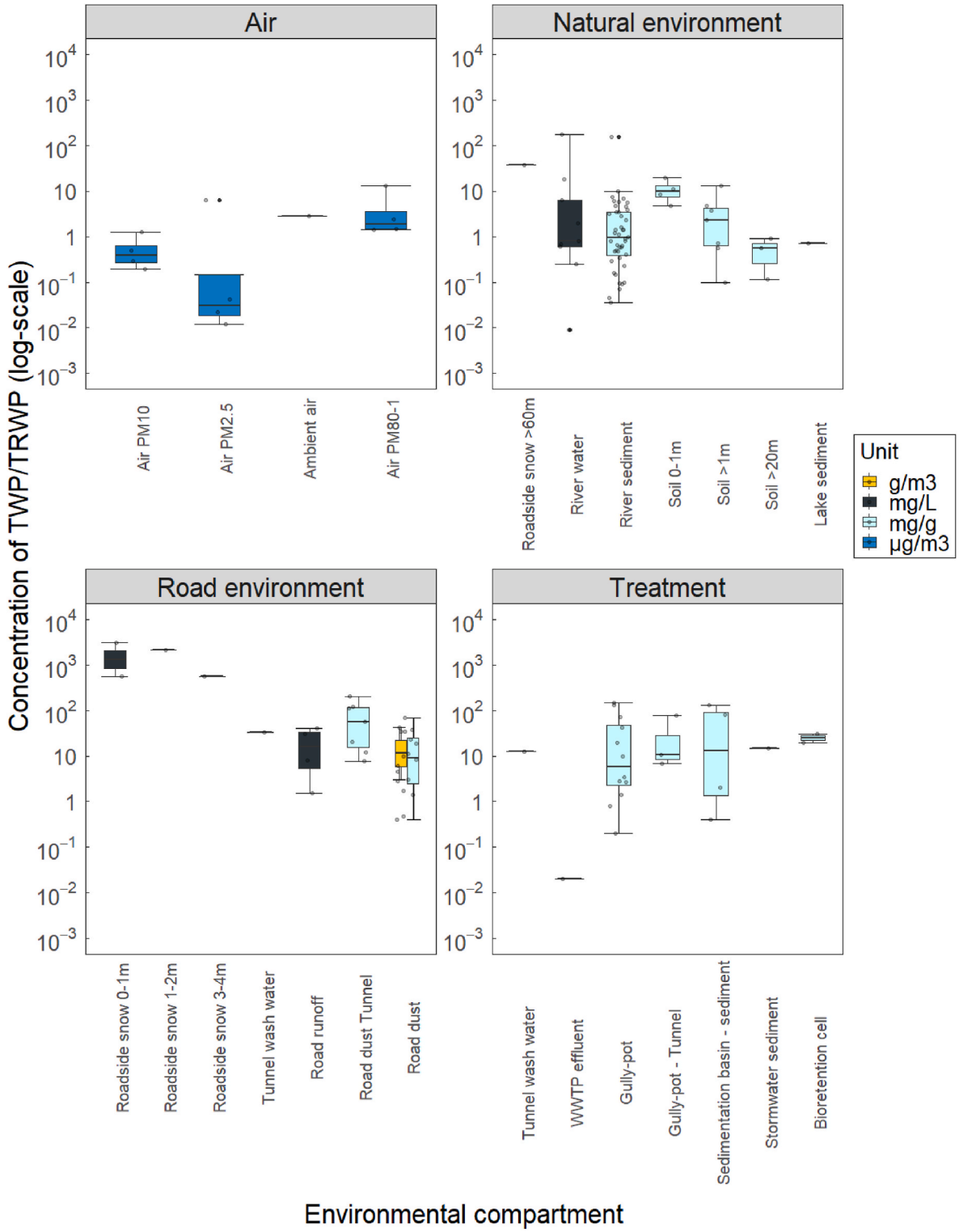

Fig. 2. Summary of the currently available mass-based concentration level of TRWP in the environment, based on average mass concentration reported in different peer-reviewed studies, as described in Table 2. In the case that no average values were reported, the minimum and maximum values (range) were applied. These values are described in Table 2.

immersing whole tires in water and then exposing aquatic organisms to the released leachate [95]. This approach solidified the idea that the impact of tire rubber exposure was attributed solely to their leachate [6, 21]. Although a range of species were exposed using standardised toxicity methods, there was little meaningful read-across (Fig. 3A). In light of microplastics acting as chemical vectors [96,97] interest in tire particles has renewed, particularly since 2018. With new analytical approaches available to better characterise exposures, the ecotoxicological focus of contemporary tire particle research has diversified (Fig. 3).

Tire particles generated in the laboratory (TWP), following environmental release (TRWP) or repurposing (ELT) are characterised

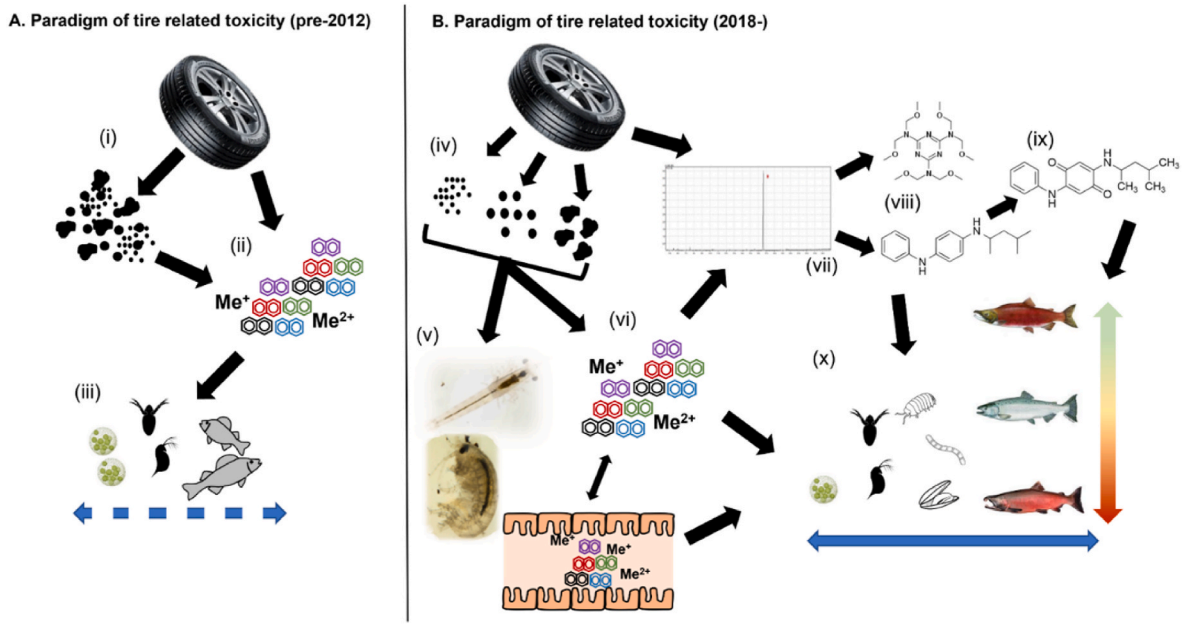

Fig. 3. Schematic representation of the development of tire particle toxicity research. (A) Prior to around 2012, research related to the toxicity of tires focused on the leachate derived from whole tires or tire granulate of unspecific size distribution (i). The leachate (ii), whilst recognised as a mixture of chemicals and compounds (metals represented as Me^{+} and Me^{2+} and organics depicted as coloured aromatic rings), was mainly treated as a singular contaminant and biological tests (iii) were predominantly conducted as standard aquatic toxicity tests to a leachate dilution series. There was minimal meaningful cross species comparison. (B) In the last 5 years, the toxicity research on tire particles has become demonstrably more complex. Particle generation either in the laboratory (TWP), following environmental release (TRWP) or repurposing (ELT, TCR) is subject to physicochemical analysis (iv). Exposure to the particles themselves has led to research into particle ingestion as an important route of entry and the potential of *in vivo* leaching within an organism's digestive tract (v). Leachates are now produced from particles (vi) under more environmentally realistic conditions. Advanced chemical analysis methods (non-target and target) are used to identify individual components in the leachate (vii). Individual chemicals found in the environment, such as 6PPD or hexamethoxymethylmelamine (viii), as well as their transformation products, such as 6PPD-quinone (ix) are now being investigated independently. A greater variety of organisms are now exposed to tire particles, leachates, individual components, or their transformation products, which represents an expansion of biological test scenarios (x). Comparisons across species and within specific taxonomic groupings are now being undertaken, as exemplified by the determination of species sensitivity of Salmonidae to 6ppd and 6PPD-quinone.

physically (particle-based) or chemically (mass-based) (Table 1, Fig. 1) alongside exposures to aquatic organisms. Leachates are now produced within exposure media, increasing environmental realism [63,98,99] rather than the forced leaching involving solvent extraction and high temperature over a short duration that were previously commonplace [6]. Exposures to leachates remain relevant, but leachates are now recognised as a complex suite of individual chemicals [100] with each chemical migrating from the polymer at its own rate and having an individual bioavailability dependent on behaviour in the media, the influence of the released chemicals upon one another (i.e. competition and complexation) and the species-specific physiological parameters at the site of uptake [9,97]. Thus, several recent studies have shown the toxicity of the leachate may be driven by just a few chemicals or groups of chemicals, e.g. benzothiazoles and aryl-amines [4], organophosphates and trace metals [98]. Identifying toxic agents amongst the thousands of chemicals within tire leachate is an ongoing process, but the additional complexities of driving age, weathering and aging can cause the release of more chemicals from TWP. In one of the few studies to compare pristine and road-worn TWP and leachates, pristine TWP was more toxic than road-worn TWP to *H. azteca* with four compounds showing markedly different concentrations, namely, 1-octanethiol, phenanthrene, anthracene and aluminium [7].

In addition to waterborne leachate exposures, the ingestion of TWP has been demonstrated in various aquatic species [9,16,101,102]. A gut retention time for TWP in *H. azteca* was established at 24–48 h [102] and while the number of ingested particles by *Gammarus pulex* was similar for MPs and TWP, the number of TWP retained in the gut was higher [103]. Considering both waterborne and dietary routes of entry means that the site of uptake for leached chemicals simultaneously becomes the respiratory surface (i.e. gills) and, given the potential for *in vivo* leaching post-ingestion, the digestive tract. Studies attempting to delineate particle and leachate effects have shown differential toxicity between the two fractions. When directly comparing particulate and leachate toxicity, the acute and chronic mortality of *H. azteca* showed distinct toxicity profiles, and reproductive output and growth were impaired at high TWP exposures [102]. Leachate was the main driver of toxicity for *D. magna* and larval zebrafish, but the toxicity resulting from exposure to nano-sized tire rubber was greater than leachate exposure alone, suggesting that particle size is also important [22].

The isolation of individual chemicals from leachate is now possible owing to non-target and target analysis (section 4.2). The identification of 6PPD (N-(1,3-Dimethylbutyl)-N′-phenyl-p-phenylenediamine) as the cause of acute mortality in coho salmon [104] has opened new avenues of research where exposures to tire-derived chemicals are conducted independent of tire particle or leachate exposure. The cross-linking agent hexamethoxymethylmelamine (HMMM) is another emergent family of tire derived chemicals that displays environmental ubiquity [105], though investigations into its toxicity are scarce. Furthermore, chemicals like 6-PPD and HMMM can be environmentally transformed, with the derived products potentially exhibiting higher levels of toxicity than the parent chemicals, as seen in the case of 6PPD and 6PPD-quinone (section 6.2).

6.2. Focus on 6PPD and its derivative 6PPD-quinone

6PPD is a commonly used antioxidant and antiozonant in vehicle tires, with a predicted proportion per total tire rubber mass of 0.4 %–2 %. 6PPD-quinone is formed due to a reaction with ground-level ozone [104]. Both 6PPD and 6PPD-quinone, which are considered emerging pollutants, have been documented in diverse environmental

compartments, including water, atmosphere, dust, and soil [13]. Recent landmark studies on the toxicity of urban stormwater runoff have revealed that 6PPD-quinone, with a very low 24h LC50 of 0.095 μg/L, may be the cause of mass mortality of Pacific Northwest coho salmon before they spawn in freshwater streams, commonly referred to as Urban Runoff Mortality Syndrome [104,106,107].

The susceptibility to this pollutant, however, is highly species-specific, and phylogenetic proximity is not a reliable predictor of reported toxicity. Laboratory studies demonstrate that 6PPD-quinone is highly toxic to coho salmon, brook trout, and rainbow trout, but moderately toxic to Chinook salmon and steelhead trout, and the least toxic to Arctic char, chum salmon, Atlantic salmon, Sockeye salmon and brown trout [12,106–109] (Table 3). Sensitive Salmonidae exhibit behaviours indicative of cardiorespiratory distress [12,106]. Exposure studies using primary cultures of rainbow trout gill cells further indicated that the toxicity of 6PPD-quinone might be caused by tissue-specific impairment of mitochondrial respiration [110], whereas exposure of juvenile coho salmon to roadway runoff revealed a potential mechanism of blood-brain barrier disruption [111]. Differences in salmon susceptibility may be caused, at least in part, by toxicokinetic factors [110]. In zebrafish, research revealed low 6PPD and 6PPD-quinone mortality, but they can induce developmental, behavioural, and cardiotoxicity at higher concentrations in larvae [112,113] and cause abnormal behaviours in adult zebrafish through changes in neurotransmitter levels [114]. Worryingly, 6PPD and 6PPD-quinone have now also been reported in human urine samples [115].

7. Regulation

Very few studies have investigated the environmental regulation of tires. Trudsø et al. [18], mapped the European regulations related to the life cycle of tires, focussing on chemicals, manufacturing, raw materials, road use, waste handling, safety and polycyclic aromatic hydrocarbons (PAHs). The life cycle of a tire consists of five overall stages: 1) raw material manufacturing, 2) product manufacturing, 3) on the road (use), 4) End-of-life tire disposal and 5) repurposing scenarios. After having first mapped what is known about the mass flow during the different life cycle stages of a tire, Trudsø et al. [18] identified key EU legislation currently in place at different life cycle stages of a tire. The subsequent analysis of the various legislative acts with regard to definitions, information and reporting requirements, (eco)toxicological thresholds and risk assessment and monitoring and enforcement revealed that no regulation directly addressed the contribution of tire particles to the environment and there is a lack of focus on the compounds that partition from the tire and disperse into the environment, their mixture effects, and the transformative products. Priority should be given to addressing these shortcomings.

The EU's REACH (registration, evaluation, authorisation and restriction of chemicals) regulation currently exempts polymers from registration. The definition of polymers covers substances taking part in the polymerization reaction, together with additives necessary to preserve, batch-impurities, reaction-by-products and catalysts. Therefore, it has been suggested that polymers are treated as mixtures in the context of hazard assessment [116]. Additionally, tires may act as carriers of other toxic contaminants [117]. REACH does not ensure transparency about which kinds of chemicals are present in a tire. It is only recently that chemical derivatives have come into focus (e.g., 6PPD-quinone (see section 6.2)). More focus is needed on the constituents of tires, their mixture effects, and their transformation products.

There is an evident need to understand both the hazards of polymeric materials and chemicals, and their transformation products. Screening a complex mixture matrix for each of the possible chemical components in particles and leachate is a challenging and time-consuming endeavour [2]. It is clearly not possible to assess the hazard for each chemical compound that can be identified in the ~120000 different tires in the EU alone. To overcome this, scientists must resort to non-standardized testing methods, as standard test methods remain inadequate for particulates [118].

Although the individual constituents are regulated under REACH regarding raw materials and production, they are not considered part of tire particle pollution, and many of the chemicals are not accounted for. Secondly, EU legislation generally makes it difficult to regulate the effects of complex mixtures. Complete mixture effects, knowledge about the environmental fate of each compound in the different life stages of a tire, and metabolites of compounds are not investigated properly to ensure an environmentally sound exposure of tire particles. Read-across from chemical risk assessment is insufficient for assessing the hazard potential of tire particles.

8. Mitigation

In 2021, the Organisation for Economic Co-operation and Development (OECD) published a report on 'textiles and tyres' and here it was highlighted that mitigation is necessary across the product life cycle, at the design and manufacturing stage, during use, at the end-of-life, and at the end of pipe capture [119]. Tire particles are a concern within

Table 3
Overview of the sensitivities of Salmonids to 6PPD-quinone.

Sensitivity	Species	Life stage	LC50 (μg/L)	Relative mortality (%)	Exposure conc. (μg/L)	Exposure time (h)	Reference
Sensitive	Coho salmon (*Oncorhynchus kisutch*)	Juvenile	0.095		0.020–0.200	24	Tian et al., 2022 [104]
	Brook trout (*Salvelinus fontinalis*)	Sub-adult (1 year)	0.59		0–20	24	Brinkmann et al., 2022 [12]
	Rainbow trout (*Oncorhynchus mykiss*)	Sub-adult (2 years)	1.00		6 and 20	72	Brinkmann et al., 2022 [12]
Intermediate	Steelhead (*Oncorhynchus mykiss irideus*)	Sub-adult (1 year)	n.d.	4–40	0.59–0.93*	24	French et al., 2022 [108]
	Chinook salmon (*Oncorhynchus tshawytscha*)	Sub-yearling	n.d.	0–13	0.59–0.93*	24	French et al., 2022 [108]
Tolerant	Arctic char (*Salvelinus alpinus*)	Sub-adult (3 years)	n.d.		20	96	Brinkmann et al., 2022 [12]
	Chum salmon (*Oncorhynchus keta*)	Prespawn adult	n.d.		2.4	24	McIntyre et al., 2021 [106]
	Atlantic salmon (*Salmo salar*)	Alevin	n.d.		0.095–12.16	48	Foldvik et al., 2022 [109]
	Sockeye salmon (*Oncorhynchus nerka*)	Sub-yearling	n.d.		0.59–0.93*	24	French et al., 2022 [108]
	Brown trout (*Salmo trutta*)	Alevin	n.d.		0.095–12.16	48	Foldvik et al., 2022 [109]

academia but have also come to the attention of new technology start-ups and the tire industry itself. Outside of academic circles, there are many notable examples to better understand and reduce the entry of tire particles and chemicals into the environment.

- Emissions Analytics are in the process of compiling a database of tire chemicals utilising two-dimensional gas chromatography coupled with time-of-flight mass spectrometry [57]. Their data indicate large differences in the chemical composition between tire manufacturers and models, and potentially identify more reliable markers for environmental detection.
- The 'Tyre Collective' is a technology start-up that aims to capture tire particles as they are created from abrasion using electrostatics and airflow to attract particles into filters [120].
- Tire manufacturers are seeking to build 'sustainable' tires with alternative materials. Continental, for example, is developing tires with rubber derived from dandelion roots, while Bridgestone Group and Nokian Tyres are attempting to manufacture rubber from guayule, a woody shrub [121].

Such steps forward are needed, and the scientific community should strive to collaborate with others, including tire manufactures, who are attempting to reduce the environmental impact of tires.

9. Conclusions and outlook

The presence, effects and solutions to tire particles in the environment could be described within the same framework as the 'Wicked Problem' proposed for microplastics [122]. While advances have been made within the various fields, it is the continued development and availability of analytical techniques that underpin our increased understanding of environmental presence and leachate release under more environmentally realistic scenarios. However, several challenges remain across the different topic areas (Table 4) and these must be prioritised as future research directions. To know how much tire particle is entering the environment, estimates of emissions need to be better related to empirical studies of tire loss, which in turn need to be related to measured data on tire particles in different environmental compartments. Following this, it is vital to understand the pathway of release – transport – fate, but the variety of analytical tools available need to provide comparable and complimentary data. The diversification of toxicological interest now covers: (i) the ingestion of tire related particles, (ii) waterborne leachate exposure, (iii) exposure to individual chemicals and their transformation products (iv). While this highlights the complexities of tire particles as a pollutant, it also provides an insight into the difficulty of regulating tire particles and their leachates holistically, rather than the current practice of different constituents being separately regulated. Mitigation efforts based on sustainability and technological solutions need scientific scrutiny as alternative materials may introduce alternative problems, as seen elsewhere [123]. Tire particles are a mutifaceted environmental challenge that will require a broad alliance of stakeholder groups to advance this field and find solutions.

CRediT authorship contribution statement

Farhan R. Khan: Writing – review & editing, Writing – original draft, Conceptualization. **Elisabeth S. Rødland:** Writing – review & editing, Writing – original draft. **Pieter Jan Kole:** Writing – review & editing, Writing – original draft. **Frank G.A.J. Van Belleghem:** Writing – review & editing, Writing – original draft. **Adrián Jaén-Gil:** Writing – review & editing, Writing – original draft. **Steffen Foss Hansen:** Writing – review & editing, Writing – original draft. **Alessio Gomiero:** Writing – review & editing, Writing – original draft.

Table 4
Overview of topics within the field of tire particles, current state, and ongoing challenges.

Topic	Current state and ongoing challenges
Nomenclature	Whilst mostly resolved with the TRWP, TWP and ELT clarification, the historical diversity may hinder accurate comparisons amongst studies.
Emissions	National emission estimates are based on two differing approaches, but analysis of country-based emission estimates revealed they are based on secondary sources (summaries), rather than measured data. There remains a need to obtain reliable emission data to know the scale of tire wear entering the environment.
Analysis and detection of tire particles	Tire particles (specifically TRWP) are detectable via a range of methodologies, but there is a lack of standardized analytical guidelines or agreement on marker selection. Variability of between commercial tires complicates the calculations from marker concentration to TRWP concentration.
Analysis of leachates	Despite the advances in non-target and target screening, a comprehensive characterisation of a tire's environmental chemical fingerprint is lacking. Challenges include instrumental and data processing complexity as well as in the lack of commercial reference standards.
Environmental presence	In addition to the analytical challenges, environmental concentrations vary between studies owing to lack of standardised sampling methodologies. Information on presence in different environmental compartments is limited as is data from different countries.
Bioavailability and toxicology	There remains a need to delineate particle- and leachate-specific effects, however, research into the toxicity of tire particles has diversified to include specific chemicals (6PPD and HMMM) and their transformation products (6PPD-quinone). Information is emerging of specific mechanisms of toxicity leading to more meaningful cross-species comparisons.
Regulation	Although the individual constituents are regulated under REACH regarding raw materials and production, they are not considered part of tire particle pollution, and many of the chemicals are not accounted. EURO7 will now include limits on tire wear.
Mitigation	Much of the efforts to mitigate tire pollution are being made by private companies and the tire industry as they seek alternative materials. It is important that such advances are not ignored by the scientific community and instead held to scrutiny.

Declaration of competing interest

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

Data availability

All data in analysis is from published work and is cited

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