

A REVIEW OF POTENTIAL PHYSICAL AND CHEMICAL MARKERS FOR TYRE AND ROAD WEAR PARTICLES

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ABSTRACT

Plastic pollution poses a substantial environmental challenge on global scale. Recently, tyre and road wear particles (TRWPs) have been recognized as a source of microplastic pollution to the freshwater environment. Whilst there is a growing concern regarding the potential environmental effects of microplastics, TRWPs are especially concerning because of the additives they have. These additives are utilised in the manufacturing of tyres; persist in the final product; become environmentally available; and may pose significant threats to an ecosystem. A current issue is the identification of specific constituents of TRWPs responsible for these threats. A comprehensive review of the existing literature is presented focusing on the physical and chemical characteristics of TRWPs with the aim to identify suitable marker(s). Wear particles derived from tyre tread possess distinctive a sausage shape that is exclusive to TRWPs. A range of chemical additives linked to tyres have been employed to quantify TRWPs, overlooking other potential sources such as brake wear and exhaust emissions. We found that significant amounts of 6PPD is used for the formulation of tyres, which is why 6PPD, and a comparatively stable transformation product 6PPD-quinone, could be used for the identification of TRWPs. We recommend that sampling and analysis methods be thoroughly documented to enhance the reproducibility.

1. INTRODUCTION

TRWPs are non-exhaust particles related to traffic, generated due to rolling resistance between a tyre tread and the surface of a road (Mattonai et al., 2022). TRWPs are one of several non-exhaust emissions that are of concern due to evidence of associated health problems and premature mortality (Boogard et al., 2023). Others are road wear and brake wear (Charron et al., 2019). When a brake is applied, brake pads enforce pressure against the rotors to slow down the vehicle and alter the vehicle's kinetic energy into thermal energy. Forced deceleration results in frictional heat, leading to wearing of the brake pads and the rotors (Güney and Ali, 2020; Hussain et al., 2017). The generation of brake wear depends upon the severity of braking, driving style, prevailing road conditions and composition of the brake system (Rahimi et al., 2021). However, brake wear emissions are predicted to decrease in future due to the emergence of regenerative braking systems (Liu et al., 202). A regenerative braking system takes the energy produced while slowing down a vehicle and uses it to recharge the

batteries of motor vehicles instead of wearing the brake pads. Hoofman et al. (2018) found that regenerative braking could reduce the wear of brake emissions by up to 66%. Similarly, Nopmongcol et al. (2017) suggested that a regenerative braking system could reduce brake wear emissions by 25%. However, the problem of TRWPs remains until this technology is globally widespread.

Road wear particles (RWPs) are tiny particles that wear away from road surfaces due to friction between a tyre's surface and that of road surface. These particles are found embedded in the tyre wear particles (TWPs), thus called TRWPs (Gustafsson and Johansson, 2012). However, there is inconsistency in the literature regarding the terms used for such particles. In this paper, the term tyre wear particles (TWPs) will be used for the particles generated in the laboratory without road surfaces whilst the term TRWPs for the particles generated with road surfaces and those found in the environment.

An increasing number of TRWPs and their persistence in the environment has gained the attention of scientists

because of the potentially adverse health and environmental impacts. The global emission of tyre wear particles is ~5,917,518 tonnes/year. The UK contributes about 1% (63,000 tonnes/year) of the total emission (Kole et al., 2017). However, this number is likely to grow due to an anticipated increase in the numbers of electric cars (e-cars). Timmer and Achten et al. (2016) and Kole et al. (2017) found that e-cars, as they are heavier, produce 20% more TRWPs. Despite this large emission rate, little information is available about their occurrence, pathways and sinks in the environment. This is because of limitations in present analytical methods, which have typically been designed for conventional microparticles. A lack of verified reference material for identifying TRWPs further hinders the validation of analytical methods. As tyres are comprised of organic and inorganic constituents, use of constituent(s) as a marker seems like a promising approach for the quantification of the TRWPs in the environment. While benzothiazole and zinc have been traditionally employed as markers for the identification of TRWPs, their widespread presence does not necessarily qualify them as suitable markers (Jeong and Woo, 2022; Luongo et al. 2016; Unice et al., 2013). Hence, it is necessary to completely understand the composition of tyres and the particles they shed so that their environmental risks may be evaluated.

Quantification of the mass concentration of TRWPs is a challenging task. Therefore, the identification of distinctive markers, to accurately assess the actual concentration of TRWPs, has become a focal point of research. This paper aims to identify and critically review the markers (physical and chemical) identified so far and propose potentially suitable markers for the identification of TRWPs.

2. CONTEXT

2.1 Production of tyres

An ongoing increase in the number of motor vehicles has led to an extensive production of tyres worldwide. Global vehicle ownership has reached 14.46 million vehicles (Luo et al., 2021) and correspondingly tyre production has increased to 17.85 million tyres in 2019 (Luo et al., 2021). The production of tyres reached 2000 million in 2021 and is likely to reach 5 billion by 2030 (Grammelis et al., 2021). China, as the largest tyre manufacturer, produced ~449 million tyres in first half of 2021 (Yang et al., 2022); thus contributed ~22% of the world's production of tyres (Mavukwana and Sempuga, 2022).

The primary function of tyres is to bear the weight of a vehicle and to grip the road while a vehicle is moving, accelerating, braking, and cornering (Wik and Dave, 2009). A tyre has a complex structure as shown in Figure 1. Briefly, the typical structure of a tyre comprises tread, crown ply (nylon), steel belt, body ply, carcass, and inner lining (Luo et al., 2021; Dobrotă et al., 2020). The outer layer, tread, contacts the road surface while driving and provides directional stability. Softeners, vulcanizing agents, plasticizers, carbon black and fillers are mixed in different proportions, to give the desired characteristics to the tyre composite.

The specific composition of each tyre type is different for required features. For example, winter tyres contain more natural rubber, as it provides grip on wet and snowy roads (Continental, 2019). The exact recipe of tyre composition is not usually publicly released by tyre manufacturers, but a typical general composition includes rubber polymers, softeners and anti-degradants (Luo et al., 2021; Dobrotă et al., 2020) (Table 1).

Kole et al. (2017) estimated that about 6 million tonnes of TRWPs are released in the environment annually world-

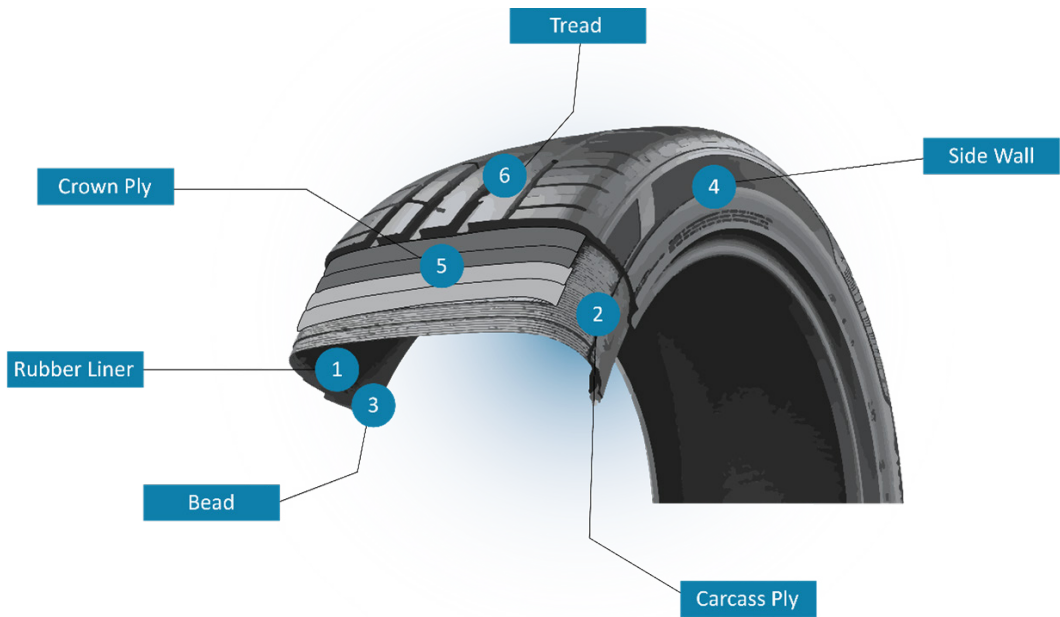


FIGURE 1: Structure of a typical tyre: 1. Rubber liner - airtight layer of synthetic rubber 2. Carcass ply- thin fibers fused within rubber 3. Bead - the boundary of tyre that contacts the rim. 4. Sidewall - vertical part of tyre that connects the tread to the bead. 5. Crown ply - provides rigid base for tyre tread. 6. Tread - designed to make contact with road and maintain optimal friction (Adapted from Dobrotă et al., 2020).

wide. The generation of TRWPs depends on a number of factors, including type and weight of vehicle, speed and road type (Table 2) (Unice et al., 2019; Kole et al., 2017; Kumata et al., 2000). This suggests that the characteristics of TRWPs are different across the world (Mennekes and Nowack, 2022). For instance, asphalt roads in European countries are reported to have a higher wear rate as compared to concrete roads (Pohrt, 2019; Gustafsson and Johansson, 2012), probably due to asphalt having particles of mineral aggregate, filler and bitumen binder (Pohrt, 2019). Bitumen particles wear away with TRWPs, increasing surface voids (Pohrt, 2019). The roughness of this texture increases with time. The rough surface area increases friction between a tyre and a road surface, leading to increased wearing of both (Pohrt, 2019). Concrete roads are made of sand and stone (granite) bound with cement (Kole et al., 2017; Lee et al., 2013) and has a comparatively smooth surface. Seasonal variations influence wear rate. Wear rate has been reported to increase up to 4% by 1°C increase in temperature. This has been confirmed by a reported 31% increased life span of passenger car tyres during the winter season (Pohrt, 2019). Similarly, wear rate has been reported to decrease during rainfall (Parker-Jurd et al., 2018). This might be due to the aggregation of particles already present on the road during rainfall, resulting decrease friction between tyre tread and the surface of a road (Changarnier et al., 2018).

2.2 Estimation of wear particles

Various emission models, employing different approaches, are used to calculate the release of TRWPs in different countries (Table 3). A commonly used approach is based on calculation of an emission factor, which is mass release of wear particles per tyre over a driven distance.

TABLE 2: Factors effecting generation of TRWPs, Adapted from Luhana et al., 2003.

Characteristics	Factors affecting the characteristics
Tyre	Size(radius/width/depth) Tyre age Tread depth Construction Tyre pressure and temperature Tyre hardness Size of tyre Tread grooves Chemical composition Rim size Tyre alignment Total distance travelled
Vehicle specifications	Vehicle type Wheel alignment Vehicle weight and distribution of load Location of driving wheels Engine power Suspension system Power/unassisted steering Axle configuration (e.g. 2 or 4 wheel drive) Electronic braking systems
Road surface characteristics	Material: bitumen/concrete/ asphalt Slope Texture pattern Texture wavelength - micro/macro/mega Presence of debris on road Porosity Condition including rutting and camber Road surface wetness Silt loading of road surface Surface dressings Temperature
Vehicle operation	Speed (average or max) Load Linear acceleration/ deceleration Radial acceleration/ deceleration Frequency and extent of braking Driving style and driving characteristics
Weather	Rain Sunlight Snow Temperature

TABLE 1: Composition of typical tyre tread and the road surface. Adapted from Sommer et al., 2018 and Krieder et al., 2010.

Particle from	Component	Percentage	Main ingredient	Characteristics
Tyre tread	Rubber polymer	40-50%	Natural rubber Isoprene rubber Butadiene rubber Styrene Butadiene rubber	- Gives elastic characteristics to the tyre - Provides directional stability
	Reinforcing agents	25-35%	Carbon black or silica	Increases the life span of tyres
	Softeners	15-20%	Humic acid (HA) oil	Helps the tyre to have grip on the road
	Vulcanizing agent	1-2%	Sulphur Zinc oxide Stearic acid	Helps in increasing the elasticity of the tyres
	Vulcanization accelerators	0.5-2%	Chemical compounds, e.g.: 2-mercapto-benzothiazole (MBT), N-Cyclohexyl-2-benzothiazole sulfenamide (CBS), N tertiarybutyl-2-benzothiazole sulfenamide (TBBS), N-Oxydiethylent-2-benzothiazole sulfenamide (MBS), Hexamethylene Tetramine (HMT), Mercaptobenzothiazole disulfide (MBTS), N,N-Dicyclohexyl-2-Benzothiazole sulfenamide (DCBS) and Tetramethyl-thiuram-Disulfide (TMTD)	- Act as a catalyst - Increases the vulcanization process
	Activators	2-5%	Zinc oxide, zinc and fatty acid	Helps in vulcanization process
	Antidegradants	1-2%	Antioxidants and antiozonants, e.g. 2,2,4 trimethyl-1,2-dihydroquinoline (TMQ), N-isopropyl-N'-phenyl-p-phenylenediamine (IPPD), N - (1,3-dimethylbutyl)- N -phenyl-p -phenylenediamine (6PPD) and N,N'-Bis (1,4-dimethylpentyl)-p-phenylenediamine (77PD)	Helps in improving durability of rubber

TABLE 3: Tyre and road wear particles in different countries, Adapted from Baensch-Baltruschat et al. 2020; Wik and Dave, 2009.

Matrix	Concentration	Country	Reference
Road dust (mg/ vehicle km)			
Road surface	72000	USA	Hopke et al. (1980)
Inside tunnel	2700	Japan	Saito (1989)
Outside tunnel	2100	Japan	Saito (1989)
20m from roadside	900	Japan	Saito (1989)
Road surface	35000	USA	Rogge et al. (1993)
Road surface	1100	USA	Reddy and Quinn (1997)
Road surface	700	Denmark	Fausser et al. (1999)
Inside tunnel	210000	Japan	Kumata et al. (2000)
Outside tunnel	124000	Japan	Kumata et al. (2000)
Road surface	56000	Japan	Kumata et al. (2002)
Calculations t/annum			
Emission factor	1327000	Germany	Wagner et al. (2018)
Emission factor	98400	Germany	Baensch-Baltruschat et al. (2020)
Emission factor	6514-7660	Denmark	Kole et al. (2017)
Emission factor	4200-6600	Denmark	Lassen et al. (2012)
Emission factor	7310	Denmark	Kole et al. (2017)
Emission factor	37646	France	Unice et al. (2019)
Laboratory wear tests	50000	Italy	Millani et al. (2004)
Emission factor	17300	Netherlands	Verschoor et al. (2016)
Emission factor	15030	Netherlands	Kole et al. (2017)
Emission factor	42000-84000	Great Britain	Kole et al. (2017)
Emission factor	20000	Australia	Millani et al. (2004)
Emission factor	294011	Brazil	Kole et al. (2017)
Emission factor	756,240	China	Kole et al. (2017)
Emission factor	292674	India	Kole et al. (2017)
Emission factor	239762	Japan	Kole et al. (2017)
Emission factor	1524740	USA	Kole et al. (2017)

Emission factors are different for different vehicle classes. For instance, Mennekes and Nowack (2022) found that a passenger car tyre loses about 117 mg of tread particles per km but this value is much higher for truck tyres. Similarly, in the study by Kole et al. (2017), the emission rate for 14 countries was estimated to range from 230,000 to 1.9×10^6 mg/capita/year. England's Environment Agency reported the average wear rate of a typical tyre of a passenger car to be 140,000 mg/metre per tyre in Great Britain (Wik and Dave, 2009). However, this wear rate has been noted to be significantly higher in city areas, owing to increased accelerating and braking (Lee and Kim, 2020; Wik and Dave, 2009). In this way, 10-40% of tread rubber (from passenger cars) is emitted into the environment in the form of wear, over the life of a tyre. Wik and Dave (2009) estimated the concentration of TRWPs at different sites (Table 3).

For Japan, it ranges from 900-210,000 mg/km. Similarly, for USA it is 1,100-72,000 mg/km. The variation in concentration of TRWPs might be due to distance from the source or due to different method used for estimation. In addition, Baensch-Baltruschat et al. (2020) calculated yearly emissions of various European and non-European coun-

tries (Table 3). The variation is probably due to different road infrastructure, different vehicle characteristics and number of cars owned by people.

A second approach is the use of material flow analysis (MFA). This method involves estimating production of tyres (by import and export) and the number of old tyres collected (Mennekes and Nowack, 2022). The average life span of a tyre is about 60,000 km before coming to an end (Mennekes and Nowack, 2022). In context of these studies, it has been found that calculation of TRWPs is based on reports and reviews instead of actual experimental work. Therefore, it is important to accurately determine the quantity and composition of emissions of TRWPs.

Furthermore, ~100 million used tyres end up as a waste globally every year and the quantity is expected to grow because of vehicle development (Wang et al., 2019). End-of-life tyres are either dumped in landfills (Gigli et al., 2019) or shredded to produce tyre crumb rubber (TCR) (Hüffer et al., 2019). TCR is produced by first removing the steel belt and then grinding the tyres (Hüffer et al., 2019). TCR is used for various applications. For example, to construct artificial turf fields in schools, colleges, practice fields and

stadiums, in mulches, to produce modified asphalt having crumb rubber, for civil engineering applications and for military installations (Benson et al., 2019; Hüffer et al., 2019). TCR may include potentially toxic constituents that possibly leach or volatilize in the environment (Sibeko et al., 2020). Therefore, human exposure to these crumb rubbers may cause various health problems; e.g. Watterson, (2017) reported that these crumb particles cause respiratory and skin problems. Similarly, Gryniewicz-Bylina et al. (2022) found respiratory problems due to inhalation of particles. According to the European Chemical Agency (ECHA), there are more than 13,000 artificial turf fields in Europe (Watterson, 2017). Millions of people are estimated to play on these artificial pitches. Compared to grass, these pitches allow people to play year-round, irrespective of weather conditions (Verschoor et al., 2016). Watterson (2017) and Benson et al. (2019) evaluated that 1,500-5,000 kg of tyre crumb is used in creating synthetic fields and ~500 kg of refill is required for each field every year, indicating that 10% of the used tyre crumb rubber from each field is released into the environment (Verschoor et al., 2016). Most of the released tyre crumb rubber is coarse in size (>50 µm) (Mattonai et al., 2022; Wagner et al., 2018).

2.3 Change in the characteristics of wear particles

Once released into the environment, TRWPs are subject to change in their physical and chemical structure due to changing environmental conditions. It has been observed that shear stress is expected to cause breakage or aggregation of TRWPs, involving both road dust and vehicle tyres (Wagner et al., 2022). Aggregation may include homo-aggregation or hetero-aggregation. In homo-aggregation, TRWPs combine with other TRWPs whilst, in hetero-aggregation, TRWPs bind with road dust and mineral particles (Luo et al., 2021). This might be due to the rough surface of the rubber particles that offers a large surface area to form aggregation (Sommer et al., 2018). The particles possess a circular cross section, enabling them to move over the surface of the road easily, thus collecting other particles (Sommer et al., 2018). Consequently, this aggregation leads to change in the density and size of the TRWPs. In addition, changing environmental conditions lead to chemical changes in the TRWPs that ultimately lead to changes in physical properties. For example, an increase in temperature leads to increased oxidation, resulting in either breaking chains or further crosslinking reaction (Wagner et al., 2018). Subsequently, cracks may develop in the structure.

2.4 Sinks and impacts of TRWPs

The size and density of TRWPs determine their fate in the environment. Being coarser in size (>50 µm), most of the TRWPs make their way to roadside soil. According to Wagner et al. (2018), about 90% of the generated particles are non-airborne and suspected to be present in roadside soil while only 10% become airborne. Järnskog et al. (2020) estimated that ~50% of TRWPs present in roadside soil wash off through precipitation, of which half reach the freshwater system and remainder goes to wastewater treatment systems (Wagner et al., 2018) and gully-pots (a part of surface water drainage system that receives sur-

face runoff (Rødland et al., 2022). Similarly, Baensch-Baltruschat et al. (2020) and Unice et al. (2019) observed that ~2% of TRWPs reach the ocean. Once in the water system, TRWPs particles may distribute in the water column depending upon the density and size of the particles. It may also depend on the density of the water (freshwater or saline water) and on the energy of the system. All these factors determine the availability of TRWPs to aquatic animals (Cunningham et al., 2022).

TRWPs may be taken up by range of organisms from algae to crustaceans and fish (Cunningham et al., 2022; Huang et al., 2021). Adverse effects have been reported in organisms and these effects might be due to physical interaction between particles and the organisms or due to the constituents (organic or inorganic) released from the particles (Wagner et al., 2018). For example, Parker et al. (2020) found ingestion of tyre particles in Striped and Spot Mullet fish. LaPlaca and van den Hurk (2020) observed mortality in mummichogs (*Fundulus heteroclitus*) and Fathead minnow (*Pimephales promelas*) following exposure to various concentrations of TRWPs ranging from 38 to 355 µm. Similarly, Cunningham et al. (2022) observed slow involuntary movements associated with the development of nervous system, change in swimming behaviour, axis malformation, hatching delays and mortality in embryos of Zebrafish (*Danio rerio*) and freshwater crustacean (*Daphnia magna*). These effects were observed following a 48h exposure to nano-sized (<1 µm) TWP (Cunningham et al., 2022). Craniofacial defects, deformed jaws, and blunt faces were observed in Fathed minnow (*Pimephales promelas*) when exposed to tyre wear particles-derived leachates. Marwood et al. (2011) reported inhibition in growth of freshwater algae (*Pseudokirchneriella subcapitata*) when exposed to different concentrations (0-10000mg/L) of leachate of TRWPs. All these factors combined make it necessary to identify constituent(s) of TRWPs that may potentially affect the ecosystem.

3. METHODS

The methodological approach for this literature review followed the PRISMA 2020 guidelines (Way et al., 2022; Page et al., 2021). In December 2023, an extensive search of database was conducted using PubMed, Scopus and the Web of Science search platforms. The search query included terms such as “elemental composition” OR “elemental marker” AND “chemical composition” OR “chemical marker” AND “physical characteristics” AND “tyre wear particles” OR “tyre and road wear particles”. The search criteria was refined to include only English language papers, with a publication date ranging from 2000 to 2023.

After the database search, 638 research articles were identified, and duplicates were removed using the reference manager (Endnote), resulting in 162 papers for screening, based on titles and abstracts. Following this screening, 40 full text articles were included for further in-depth analysis. Preference was given to papers explicitly discussing the physical and chemical characteristics of TRWPs. Subsequently, two articles were excluded from the analysis as they did not clearly discuss the characteristics of TRWPs.

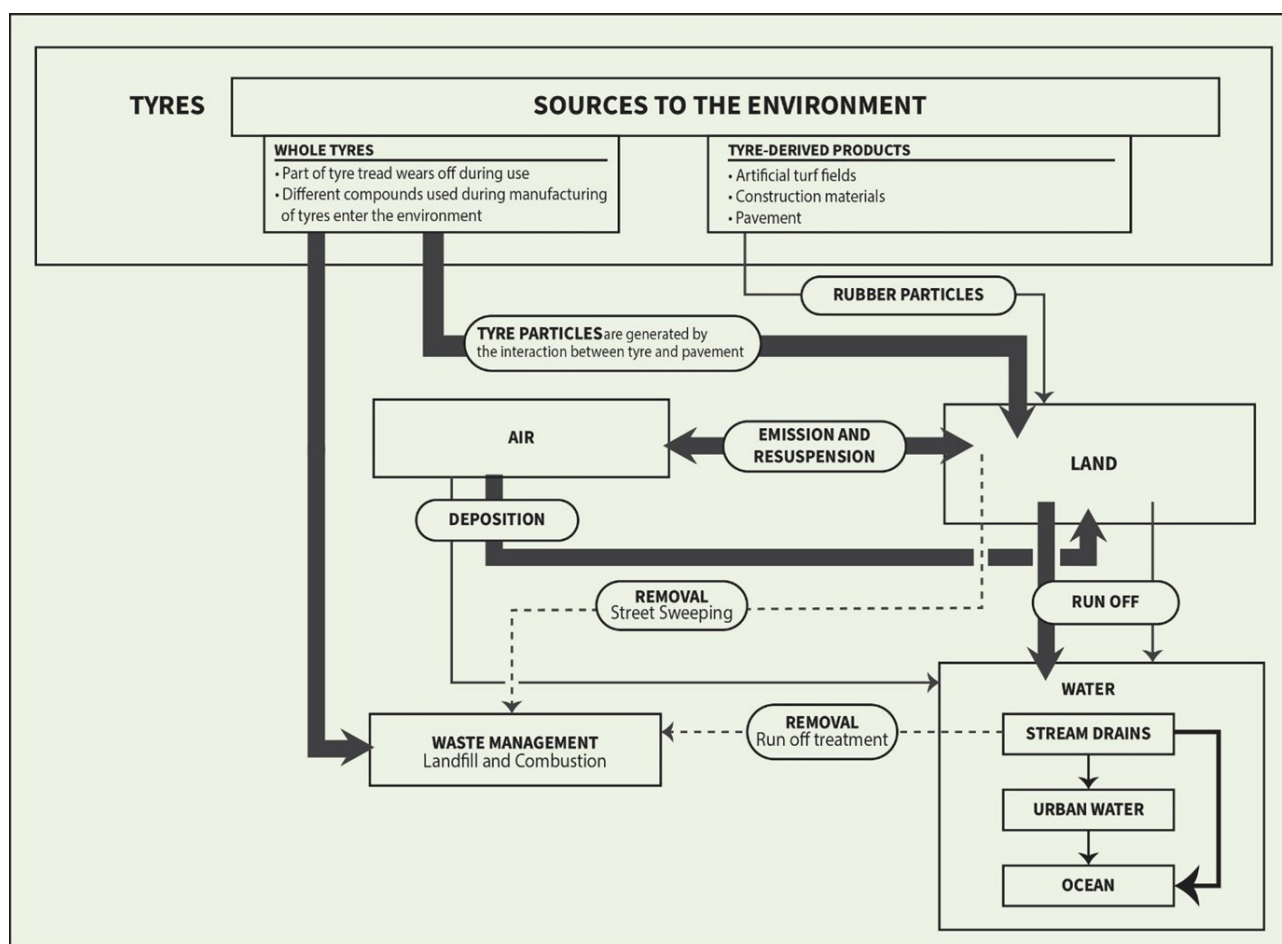


FIGURE 3: Transportation pathways of tyre and road wear particles in different environmental matrices (adapted from Mayer et al., 2024).

It was noted that the articles chosen for analysis primarily focused on particulate matter. It is important to highlight that the majority of TRWPs are typically larger. Comprehensive database search also highlighted that most of the studies on chemical characterization of TRWPs focused on vulcanizing agents and antidegradants, which is justifiable considering that ~1-3% and ~1-5% of tyre weight is attributed to protective molecules and vulcanizing agents respectively. To achieve an extensive understanding, a subsequent search using the Web of Science search engine was conducted during January 2024, without constraints on the publication date. This expanded search incorporated additional terms such as fillers, antioxidants and vulcanizing agents.

4. RESULTS AND DISCUSSION

4.1 Physical characteristics

For several years, research has focused on the identification of marker(s) for TRWPs, to evaluate the associated environmental risks. Notably, in the literature, physical characteristics have been employed for the identification. In this context, particles arising from the interaction between tyre and the surface of a road typically vary in size from a few nanometres to micrometres (Alves et al., 2020). Wear particles from tyres are produced either by tangential forces between a road surface and a tyre tread or by

volatilization (Alves et al., 2020). The initial mechanical process primarily generates coarse particles (Alves et al., 2020). For instance, Kreider et al. (2010), Kwak et al. (2014) and Knight et al. (2020) reported wear particles in the range of 50µm to 100µm. Thermo-mechanical processes also lead to generation of extremely fine aerosol particles. Mathissen et al., (2011) hypothesized that the local hot spots on the surface of a tyre tread attain elevated temperature while driving, leading to evaporative loss of volatile content of the tyre tread. Additionally, it has been noted that ultrafine wear particles are released at higher slip angle and increased speed (Alves et al., 2020).

TRWPs, based on their morphology, have been characterized into four groups. The first category comprises sausage-like/cigar-like or tube-shaped particles, commonly identified as representative forms of TRWPs (Kovochich et al., 2021; Yan et al., 2021; Kwak et al., 2014). Kovochich et al (2021) and Park and Lee, (2018) reported sausage shaped wear particles with encrustations from the road surface. Knight et al. (2020) also identified TRWPs from the roadside based on the sausage shaped appearance. These particles typically maintain their original shape in the absence of secondary wear processes (Yan et al., 2021). The second type consists of spherical particles, originating from the secondary wear processes (Yan et al.,

2021; Kwak et al., 2014). Initially, long particles are generated and later broken into smaller fragments. These particles form through shear forces resulting from friction (Yan et al., 2021; Kwak et al., 2014). Subsequently, secondary wear processes contribute to smoothing the surface of these particles (Yan et al., 2021; Kwak et al., 2014).

A third type of TRWPs consist of irregular shapes, likely formed due to intense wear processes resulting from high friction situations such as severe cornering and braking situations (Kwak et al., 2014). A fourth type is of small spherical clusters, as reported by Park and Lee (2018), with diameters <50nm. These clusters may form aggregate or attach to larger particles, making it challenging to categorize based on apparent size (Dahl et al., 2006).

Although a sausage-shaped assembly is distinctive of TRWPs, other shapes, such as spherical, irregular, and spherical clusters are not exclusive to TRWPs. These shapes may also originate from different sources, such as the wearing of brake pads and soot particles. This suggests that relying only on physical characteristics may be inadequate for identifying TRWPs.

4.2 Rubber as a marker

Given that 40-50% mass of a tyre consists of rubber polymer (Table 1), scientists found it suitable for the identification of TRWPs in environmental matrices. For instance, based on the combined content of natural rubber (NR) and styrene butadiene rubber (SBR), the reported concentration of TRWPs in road dust ranges from 23.2 to 55.0 × 104 µg/g (Miller et al., 2022). Similarly, More et al. (2023), Miller et al. (2022) and Müller et al. (2022a) observed 4-vinylcyclohexene (4-VCH) and dipentene (DP), pyrolysis products of styrene butadiene rubber (SBR) and natural rubber (NR) respectively. It is important to note that *Hevea brasiliensis* is a primary contributor of natural rubber (Venkatachalam et al., 2007). However, studies using natural rubber as a marker for TRWPs often overlook the proximity of vegetation to rule out the potential contribution from other sources of natural rubber.

Rubber undergoes vital modification to customize its properties for the production of tyres. This involves the addition of various additives to achieve the desired characteristics, a procedure termed as compounding (Rodgers and Waddell, 2005). However, additives are not chemically bonded to the polymer material, enabling them to enter the environment and potentially serve as markers for the identification of TRWPs (Johannessen et al., 2021). These additives include inorganic and organic substances. For example, metals, aliphatic and aromatic compounds, anti-oxidants/antiozonants, and vulcanizing agents.

4.3 Elemental composition

Zinc, in the form of zinc oxide, is added to tyres to help the vulcanizing process (see section 4.6) and accounts for ~1% mass of the tyre tread (Unice et al., 2013). Therefore, zinc has consistently been used as a key elemental marker for identifying TRWPs in environmental samples (Beddows et al., 2023; Beji et al., 2023; Müller et al., 2022a; Rausch et al., 2022; Choi et al., 2020; Kovoichich et al., 2021; Alves et al., 2020; Klöckner et al., 2019; Dall'Osto et al., 2014; Kwak

et al., 2013; Adachi and Tainosho, 2004). For instance, Beauchemin et al. (2021) recorded 1,258 mg Zn/ kg in a road dust sample, using inductively coupled plasma mass spectrometry (ICP-MS) as the analysis technique. However, Beauchemin et al. (2021), Ghaidan et al (2019) and Hulskotte et al. (2014) reported the presence of zinc in the wear from brake pads.

Cobalt (Co), titanium (Ti), lithium (Li), and chromium (Cr) serve as catalysts in the transformation of butadiene rubber into polybutadiene rubber (O'Loughlin et al., 2023). Barium (Ba), in the form of barite (BaSO₄), serves as a filler to enhance the ageing resistance of tyres (O'Loughlin et al., 2023). However, metals identified in TRWPs may have multiple sources on roads. For example, Co, Ti and Li are used in the engines of electric cars (Iglesias et al., 2020). Cr has been reported to be in yellow road paints (Adachi and Tainosho, 2004). Similarly, Ba is also found in paints and brake materials (Adachi and Tainosho, 2004). Therefore, Klöckner et al. (2020) raised concerns about the use of elemental constituents as markers for TRWPs and suggested considering organic constituents for their identification.

4.4 Aliphatic and aromatic compounds

Aliphatic hydrocarbon, characterized by the presence of linear or branched carbon chains, are considered essential in the formulation of tyres (Küçükosman et al., 2023). This is because hydrocarbons shield the rubber of tyre from cracking induced by UV light. Therefore, Alves et al (2020) suggested the use of aliphatic compounds (>C₃₅) for the identification of TRWPs. However, reliance on aliphatic compounds for the identification of TRWPs is questionable owing to its ubiquitous presence in gasoline. Gasoline, commonly referred to as petrol, is used as a fuel for combustion engines (Nzila, 2018). Gasoline is a liquid blend of aliphatic hydrocarbons obtained through the fractional distillation of crude oil (Vulimiri et al., 2017). Crude oil consists of a mixture of various molecules having different densities (Hsu and Robinson, 2017). The mixture is heated to extract individual fractions based on their boiling points (Hsu and Robinson, 2017). It includes heating a mixture to produce vapours which are subsequently cooled and condensed to separate the individual liquid (fractional distillation) (Hsu and Robinson, 2017). Burning of gasoline/petrol in combustion engines emits aliphatic hydrocarbons as part of the combustion process.

Aromatic hydrocarbons are groups of organic compounds that contain one or more benzene rings. These compounds are characterized by having alternating single and double bonds in their ring structure, resulting in unique stability (Küçükosman et al., 2023). Due to the stability, PAHs have been used in the formulation of tyres as softeners. Previously, polycyclic aromatic hydrocarbons (PAHs) associated with TRWPs have also been of interest. PAHs have been reported to be in fine or ultrafine TRWPs. For instance, Sharma, (2010) used phenanthrene (Phe), anthracene (Anth) and range of other PAHs to evaluate the emission of TRWPs and reported 378ng/tyre/km of PAH in PM₁₀ particles. In 2003, tyres were alleged as a major contributor of PAHs to the environment. The potential triptogenic effects (mutagenic, carcinogenic and teratogenic) of PAHs

(Yao et al., 2024) drew the attention of the European Union and subsequently it banned the use of high aromatic oils for use in tyres, as they contain PAHs (Kreider et al., 2010). Recently, Alves et al. (2020) reported PAHs in TRWPs. This might be coming from petrogenic sources. PAHs and their derivatives primarily result from the pyrolysis of fuels within motor vehicle engines (Yao et al., 2024; Ogbunuzor et al., 2020). On the surface of soot, formed during combustion, smaller PAH molecules can undergo further reactions, leading to the formation of larger and complex PAH molecules (Yao et al., 2024). They may also adsorb onto the surface of particulate matter (Ogbunuzor et al., 2020).

Considering the widespread presence of aliphatic and aromatic compounds in fuel and subsequent use as markers for the estimation of gasoline emission, these compounds may not be suitable for the identification of TRWPs.

4.5 Antioxidants/antiozonants

Antioxidants/antiozonants play a vital role in inhibiting or slowing down the degradation process of tyre rubber due to oxidative aging (Sharj-Sharifi et al., 2020). Antioxidants/antiozonants function by reacting with and neutralizing the free radicals formed during oxidative aging. This process is triggered when rubber is subjected to environmental stress factors such as light, mechanical shear and heat (Sharj-Sharifi et al., 2020). Frequently used antioxidants are N-phenyl-N'-(1,3-dimethylbutyl)-phenylenediamine (6PPD) and 2,2,4-Trimethyl-1,2-Dihydroquinoline (TMQ) (Zhang et al., 2023; Zhao et al., 2023; Hiki and Yamamoto, 2022; Klöckner et al., 2021). It has been estimated that ~54% of the yearly production of 6PPD is used as a vulcanizing agent in tyres (Zhou et al., 2023), suggesting its widespread presence in most tyres.

In static conditions, 6PPD gradually comes to the surface of a tyre or TRWPs and reacts with oxygen or ozone, creating a protective layer on the rubber surface (Sharj-Sharifi et al., 2020). The protective film effectively prevents ozone from infiltrating and interacting with the rubber (Sharj-Sharifi et al., 2020). This process of moving an antiozonant (i.e. 6PPD) to the surface of a tyre or wear particles is termed as blooming (Sharj-Sharifi et al., 2020). 6PPD may also leach into the environment and become environmentally available (Varshney et al., 2022). For instance, Hiki and Yamamoto, (2022) observed 45-1175ng of 6PPD in one gram of road dust. In a study by Klöckner et al., (2021), the presence of 6PPD was documented in road dust. Klöckner et al., (2021) collected road dust samples from a tunnel having an 80km/h speed limit. However, their sampling methodology was not discussed, compromising the reproducibility of method to verify the results. It is also necessary to analyse TRWPs to confirm the source of 6-PPD. For this reason, Unice et al., (2015) formulated an industry standard test tyre using 6PPD as an antioxidant. TWPs from a test tyre were produced through cryogenic milling, underwent weathering and were subjected to analysis using liquid chromatography-mass spectrometry (LC/MS/MS) in the mode of electrospray ionization (ESI). Analysis confirmed the presence of 1,3-dimethylbutylamine (1,3-DMBA), 4-aminodiphenylamine (4-ADPA) and 4-Nitrosodiphenylamine (4s DPA), aligning with the findings of

Zhaou et al (2023). However, tyre formulation at pilot scale may not accurately reflect the composition of wear particles present in the natural environment.

Fohet et al. (2023) exposed TRWPs (collected directly from the tyres on road) to natural aging conditions for a period of three months and found that the concentration of 6PPD decreased by 98%. It was also found that 6PPD ($C_{18}H_{24}N_2$) transformed into 6PPD-Q ($C_{18}H_{22}N_2O_2$) after 21 days of aging, suggesting that the composition of TRWPs may help in determining the age of the wear particles and the corresponding effects on the environment.

Varshney et al., (2022) found that 6PPD might negatively affect the neural system of larvae of zebrafish. Similarly, 6PPD-Q may induce mortality in Coho salmon (*Oncorhynchus kisutch*) (Greer et al., 2023). However, whilst evaluating the effects of TRWPs owing to its constituents, other possible sources of those additives should also be considered. For example, 6PPD has reported use in hair dyes and cosmetic products (Du et al., 2022).

Zhang et al. (2023) analysed the surface water samples for the targeted study of 6PPD and its transformation compounds. Water samples were supplemented with internal standards to improve the extraction efficiency and selected compounds were extracted using methanol. 6PPD-Q (maximum 1562 ng/L), along with 6PPD (max 7.52ng/L), was observed in the analysis. This not only represents the transportation of 6PPD to water channels through runoff but also indicates the potential transformation product, which is 6PPD-Q. Zhang et al. (2023) reported the presence of other potential transformation products of 6PPD namely DPPD (N,N'-Diphenyl-1,4-phenylenediamine), IPPD (N-Isopropyl-N'-phenyl-1,4-phenylenediamine), DTPD (N,N'-Bis(methylphenyl)-1,4-benzenediamine), because of the structural similarity to 6PPD. However, these identified compounds cannot be exclusively attributed to TRWPs for many reasons. Firstly, tyre wear particles have not been used as a reference, raising concerns about the reliability of the study. Secondly, the study has not considered the surrounding non-point sources of the identified compounds, indicating that the sources of the compounds has not been thoroughly investigated. Thirdly, the internal standard used may have impurities, which may cause false positive results.

Therefore, to understand the transformation mechanism of 6PPD, different researchers conducted in vitro studies. For example, Zhao et al., (2023) subjected slides, coated with industrial grade 6PPD (98% purity), to gas-phase ozone to evaluate the transformation products. Several transformation products have been identified, using liquid chromatography- tandem mass spectrometry (LC-MS/MS), such as 1,3 DMBA, 4-ADPA, 6-PPD, 4-Nitrosodiphenylamine (4s-DPA), 4-Nitrodiphenylamine (4-NDPA) and 4-Hydroxydiphenylamine (4-HDPA). For environmental applicability, road runoff samples were also evaluated; 1,3-DMBA was highest (2200ng/L) followed by 4-HDPA (150ng/L), 6PPD-Q (140ng/L), 4ADPA(110ng/L). Seiwert et al. (2022) also reported similar transformation products in aqueous phase reaction with different relative proportion, suggesting that TRWPs may be a source of these compounds in the environment. However, most of the observed transformation products of 6-PPD have various additional sources. For ex-

ample, 4-ADPA has reported use as precursor in lubricants and fuel, 4s DPA is used during production of polyvinylchloride (PVC) to inhibit polymerization reactions (Fohet et al., 2023). Therefore, 4s DPA and 4-ADPA may have multiple transformation pathways than 6PPD. Furthermore, relative high proportion of 1,3-DMBA in TRWPs and that of runoff samples indicates significant contribution from tyres to the collective environmental concentration of this compound. However, further research is required to assess the stability and significance of this component.

It is to be noted that another antioxidant that has been used in tyres, 2,2,4-Trimethyl-1,2-Dihydroquinoline (TMQ), has not been extensively examined in the existing literature.

4.6 Vulcanizing agents

Vulcanization, also known as crosslinking, is a crucial process in the tyre industry (Lubura et al., 2023; Marković et al., 2020). During vulcanization, the rubber compound transforms into a resilient product known as vulcanized rubber (Marković et al., 2020). This is achieved through sequential and simultaneous alterations of both chemical and physical characteristics of the rubber (Marković et al., 2020). The fundamental aspect of vulcanization involves establishing cross-linkages between rubber macromolecules, resulting in the formation of a three-dimensional network within rubber structure (Marković et al., 2020). The components of a cross-linked network include physical bonds (non-covalent bonds), such as hydrogen bonds, among individual macromolecule (Lubura et al., 2023; Marković et al., 2020). Interactions, including polar or dispersive forces, play a crucial role in the formation of the network (Lubura et al., 2023; Marković et al., 2020). Non-covalent interactions give flexibility and dispersive forces enhance the mechanical strength of the rubber, all crucial for the performance of a tyre (Marković et al., 2020).

Various vulcanizing agents are used to aid in the formation of chemical bonds within rubber structure. Three of the frequently used vulcanizing agents are N-cyclohexyl-benzothiazole-2-sulfenamide (CBS), benzothiazole (BT), and 1,3-Diphenylguanidine (DPG) (Weyrauch et al., 2023), as shown in Figure 3. However, all of the mentioned compounds are unstable in environmental settings (Fohet et al., 2023). It is unclear in the literature, whether CBS is a transformation product of benzothiazole or its parent compound. Fohet et al. (2023) stated that CBS transforms into benzothiazole (BT) immediately after the making of tyres (Fohet et al., 2023). Weyrauch et al. (2023) and Unice et al. (2015) reported a negligible amount (0.04%) of CBS in the extract of the particles and attributed it as a primary compound in the formulation of tyres. Zhang et al. (2018) reported various transformation products of benzothiazole and classified CBS as one of the transformation products of BT. The difference in the behaviour of CBS might be due to variations in analytical approaches and instruments used for the analysis. For instance, Weyrauch et al. (2023) used methanol for extraction of constituents from wear of tyres and used ultra performance liquid chromatography-high resolution mass spectrometry (UPLC-HRMS) for the analysis. Unice et al. (2015) used toluene as an extraction solvent and quantified the extracts using liquid chromatogra-

phy with tandem mass spectrometry (LC-MS/MS), implying the need to standardize the methodological approaches.

Similarly, BT has been widely reported in different environmental samples (Weyrauch et al., 2023). For instance, Knight et al., (2020) reported N-cyclohexyl-2-benzothiazolamine (NCBA), a derivative of benzothiazole, in drainage sumps in the United Kingdom. Wet sediments were subjected to analysis using gas chromatography mass spectrometry (GC/MS) (Knight et al., 2020). However, the specific ionization mode employed was not reported in the study. The ionization mode in analytical equipment such as GC/MS may influence the selectivity and sensitivity of the analysis of the environmental samples (Amirav et al., 2020). This could potentially impact the reproducibility and interpretation of the results (Amirav et al., 2020).

2-hydroxy benzothiazole (2-OH-BT), another derivative of BT, has been recognised as a product resulting from the oxidation of BT (Zhang et al., 2018). Laboratory studies have confirmed that the elevated temperature leads to the conversion of BT into 2-OH-BT (Borowskaa et al., 2016). Consequently, 2-OH-BT may originate from the oxidation of BT during driving, from high-speed friction (Zhang et al., 2018). However, BT and its derivatives have also been found in indoor dust as reported by Wang et al. (2013). Additionally, these compounds have been reported in textile industries (Avagyan et al., 2015).

BT has various applications (Kloepfer et al., 2004). For instance, it has reported use as fungicide and biocide (Luongo et al., 2016). Fungicides, a type of pesticide, are considered a crucial element in managing pests and diseases in horticulture (Wightwick et al., 2010). This occurs because fungal species, such as botrytis and downy mildew, can damage crops, making them unfit for sale (Wightwick et al., 2010). Nevertheless, eliminating most fungal diseases proves challenging, as outbreaks can persist over multiple growing seasons despite efforts to eradicate them (Wightwick et al., 2010). This occurs because spores may become dormant during harsh environmental conditions and start growing again when conditions become favourable (Wightwick et al., 2010). These devastating impacts of fungal diseases led to the development of fungicides. For example, experiments carried out in the United Kingdom revealed that the application of fungicide to winter wheat led to a yield increase of up to 89% (Wightwick et al., 2010). Similarly, biocides are biologically active substances employed to prevent the activities of infectious agents. These substances have various applications, ranging from maintaining hygiene in hospitals to agriculture practices, serving purposes such as disinfection and quality preservation (Kähkönen and Nordström, 2008). Additionally, these substances have frequently been detected in wastewater (Asimakopoulou et al., 2013), municipal wastewater (Kowalska et al., 2019) and household wastewater (Avagyan et al., 2015) unaffected by road runoff (Luongo et al., 2016). The extensive use of benzothiazole and its derivatives across diverse fields suggests that it may not serve as a suitable marker for identifying TRWPs.

1,3-diphenylguanidine (DPG), a bicyclic amine, is commonly used in the production of rubber as an accelerator of vulcanization. It has been associated with the pollution of

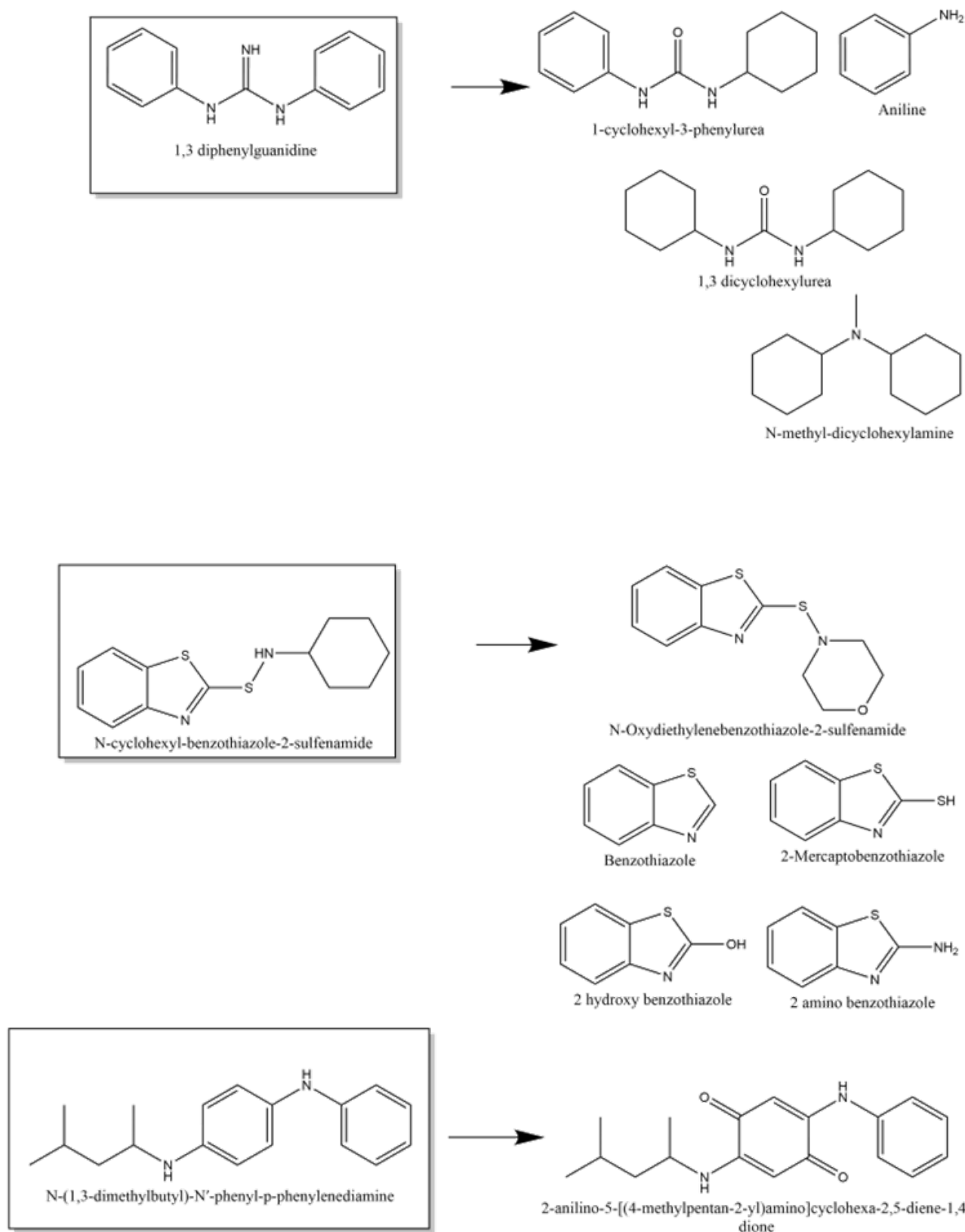


FIGURE 3: Transformation products of 1,3 Diphenylguanidine (DPG), N-cyclohexyl-benzothiazole-2-sulfenamide (CBS) and N-(1,3 dimethyl-butyl)-N'-phenyl-p-phenylenediamine (6PPD). Made by using ChemDraw software.

TRWPs (Müller et al., 2022). Johannessen et al. (2021) reported DPG in the surface water that received storm-water runoff from highways in µg/L. Moreover, Peter et al. (2018)

evaluated the leachate of tyre wear particles for DPG. Numerous compounds structurally similar to DPG were detected in the leachate, suggesting that DPG may transform

into different constituents (Peter et al., 2018). These transformed constituents include 1,3 dicyclohexylurea (1,3-DCU), N-methyl-dicyclohexylamine and N, N'-dicyclohexyl-2-benzothiazole sulfonamide (DCBA) (Peter et al., 2018). Other than these compounds, Seiwert et al. (2020) reported dicyclohexylamine in road runoff. Dicyclohexylamine is likely to be a product of hydrolysis of DCBA. DCBA consists of two cyclohexylamine groups attached to benzothiazole sulfonamide. Hydrolysis is considered to break down the bond between them, releasing dicyclohexylamine. (Peter et al., 2018). However, due to high vapour pressure and half-life of 2.9h dicyclohexylamine readily reacts with oxidizing agents (Johannassen et al., 2021), suggesting that this compound is not an ideal marker for the identification of TRWPs. Furthermore, aniline compound is also considered to be a typical transformation product of DPG (Peter et al., 2018; Unice et al., 2015). Aniline is also used as anti-knock agent in petrol (Viayna et al., 2021). Engine knocking, also known as knocking combustion, occurs when the air-fuel mixture in an internal combustion engine ignites spontaneously before the engine spark plug fires (Wang et al., 2017). This unintended ignition causes pressure waves that collide, leading to a distinctive knocking sound. Engine knocking may cause damage to the engine, causing decrease in the overall performance. Anti-knock agents, such as additives in fuels (e.g Aniline), are used to minimize knocking. These agents alter the combustion characteristics of the fuel, raising its resistance to spontaneous ignition and help to maintain engine operations (Wang et al., 2017).

Overall, it has been found that various constituents of tyre have been used for the identification and quantification of TRWPs in the environmental samples. However, most identified constituents are not ideal for this purpose. For examples, aliphatic and aromatic hydrocarbons that have been associated with TRWPs have origin from exhaust emissions as well. Similarly, benzothiazole and its derivatives have widespread use ranging from textile sector to hospitals. However, tube shaped/sausage shaped particles is an exclusive physical feature of TRWPs. Similarly, most of the world production of 6PPD is used in the manufacturing of tyres, making it somewhat suitable for the identification of TRWPs in the environment.

5. CONCLUSIONS

It is evident that identifying a single effective marker for quantification of TRWPs poses a considerable challenge. Consequently, a suite of physical and chemical markers could be used to enhance identification, quantification and impact assessment. For instance, particles having a sausage-shaped assembly could be used for the identification of TRWPs, since this shape is distinct to wear particles of tyres. As section 4.1 details the variation in shape of TRWPs, from elongated particles to spherical ones, spiking experiment should be carried out to evaluate the inadequate representation of particles other than sausage shaped. Furthermore, whilst using physical characteristics for the identification of TRWPs, zinc could function as a confirming marker to verify if the particles originated from the wearing of tyres. Similarly, 6PPD and its comparatively

stable transformation product 6PPD-Q could be used for the identification and quantification of TRWPs as more than half of the global production of 6PPD serves as an antioxidant in tyres, establishing tyres as a primary source of this compound. However, when evaluating the impact of 6PPD, it is important to consider the potential overrepresentation of this compound, recognizing that the prevalence might influence the overall representation.

Furthermore, this review of literature identified several limitations in identifying a marker for TRWPs that must be addressed when quantifying them:

- The reporting of sampling methods across studies showed significant variability. For instance, some studies have not documented the sampling method at all. The lack of comprehensive documentation regarding the sampling methods raises questions about the reliability of the collected data. Transparent reporting of sampling methodologies is important to enhance the reproducibility of the studies investigating TRWPs in the environmental matrices.
- Most of the studies included in this review did not explicitly disclose the ionization techniques used in conjunction in mass spectrometry. The absence of this vital information poses challenges for the reproducibility of the method by other researchers, as the ionization step is a fundamental aspect of the mass spectrometry process. This involves converting sample molecules into ions, which can then be separated and detected based on their mass to charge ratio (Awad et al., 2015).
- Studies focusing on the analysis of the environmental samples for targeted markers of TRWPs have not incorporated laboratory-generated tyre wear particles as a standard. This omission raises concerns regarding the potential influence of other environmental sources, as the use of laboratory generated particles serves as a critical benchmark and rules out external contribution. Incorporating such standards is essential for ensuring specificity and reliability of the identified markers in the environmental samples.

6. RECOMMENDATIONS

Based on the findings of thorough literature review, there is an urgent need to advance the future research in terms of better understanding of TRWPs. Therefore, following recommendations are proposed:

- The use of multiple thermo-analytical methods by different researchers features the inconsistency for analysing TRWPs in literature. This highlights the importance of standardizing the analytical instruments for the comparability of results.
- Artificial weathering experiments, in literature, has been conducted at 60°C; however, Kreider et al. (2010) reported the max temperature at contact point of tread and road surface to be ~180°C. Thus, future studies should also evaluate the composition and transformation products at higher temperature.
- Most of the studies have analysed targeted constituents of TRWPs without considering the possibility of

wide range of substances. Therefore, subsequent work should consider studying broader spectrum of the constituents.

- Transformation products of 6-PPD should be thoroughly investigated in aquatic ecosystem so that the potentially harmful effects and ecological risks can be assessed.
- A relatively high concentration of 1,3-DMBA in the leachate of TRWPs shows its significance. Further work is required to test if it is a degradation product of 6-PPD or a parent compound of other constituents.
- Much information about the antioxidant, TMQ, could not found in the literature, suggesting further analysis of this compound for evaluating its environmental behaviour.
- Researchers/engineers should also focus on formulating tyres that minimize TRWPs.

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