

# Screening of Tire-Derived Chemicals and Tire Wear Particles in a Road Tunnel Wash Water Treatment Basin

Sondre Meland,\* Gina Marie Granheim, Jan Thomas Rundberget, and Elisabeth Rødland



Cite This: *Environ. Sci. Technol. Lett.* 2024, 11, 35–40



Read Online

ACCESS |



Metrics & More



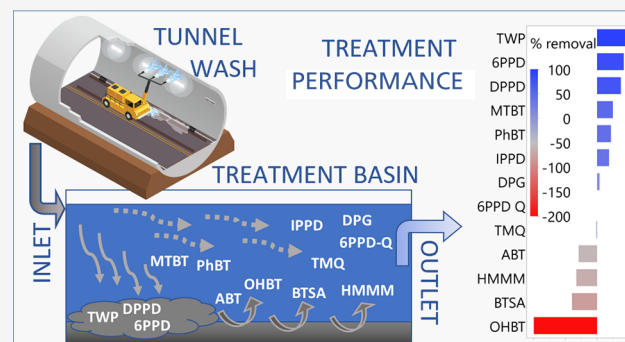
Article Recommendations



Supporting Information

**ABSTRACT:** Road tunnels play a crucial role in modern road networks, in both urban and non-urban areas, and necessitate frequent cleaning and washing due to the harsh tunnel environment. As a result, road tunnels are considered hot spots for the emission of road-related pollutants into the environment. In this study, we conducted extensive measurements of tire wear particles (TWP) and 18 tire-derived chemicals (TDCs) during the washing process, throughout a 21-day treatment period in a rectangular sedimentation basin in concrete, and during the discharge of treated wash water. Our key findings indicate that TWP are effectively retained in the sedimentation basin, demonstrating that simple mitigation measures can prevent their release near the source. However, several TDCs displayed high concentrations, mobility, and leachability, leading to inadequate retention in the basin. Moreover, some TDCs exhibited negative treatment performance, resulting in higher concentrations in the treated wash water than in the untreated wash water. Importantly, our findings can be applied to not only tunnel wash water but also normal road runoff, as sedimentation basins are widely used in many countries. This study provides a novel and significant contribution to the evolving understanding of the presence and fate of TWP and TDCs in the environment.

**KEYWORDS:** road runoff, stormwater treatment, tunnel wash water, 6PPD, 6PPD-quinone, benzothiazoles, tire chemicals, diphenylguanidine



## INTRODUCTION

In recent years, tire wear particles (TWP) have attracted attention as part of the broader category microplastics.<sup>1,2</sup> Efforts have been made to assess the emissions, fate, and toxicity of TWP in various environmental compartments such as air, soil, water, and snow.<sup>3–6</sup>

During the assessment of the occurrence and implications of TWP in the environment, there has been growing interest in understanding the emission and fate of the chemical additives present in tires.<sup>7,8</sup> In addition to natural rubber (NR) and synthetic rubber polymers [e.g., butadiene rubber (BR) and styrene butadiene rubber (SBR)], trace amounts of chemical additives (5–10% mass) used during tire production also play a crucial role in determining the properties and performance of different tire types and tire brands.<sup>9,10</sup> These tire-derived chemicals (TDCs), such as preservatives, antiozonants, antioxidants, desiccants, plasticizers, and processing aids, contribute to important characteristics like grip under different weather and road surface conditions, longevity, fuel and energy efficiency, and external noise reduction.<sup>11</sup>

A recent study detected a total of 214 organic chemicals in tire wear particles.<sup>12</sup> Of these chemicals, 145 were classified as leachable, and several are probably mobile in aquatic environments. 6PPD [*N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phe-

nylenediamine] and its transformation product, 6PPD-quinone (6PPD-Q), are two TDCs that have attracted a great deal of attention after being identified as the main suspects causing high mortality in coho salmon (*Oncorhynchus kisutch*) returning for spawning in urban streams.<sup>13</sup> The presence of pollutants, including TWP and TDCs, in urban stormwater and road runoff inevitably poses a significant environmental threat to water quality and aquatic life.<sup>14–17</sup>

One closely related concern is the discharge of tunnel wash water. Due to the harsh conditions in tunnels, regular washing is necessary to ensure visibility, traffic safety, and the longevity of technical equipment.<sup>17</sup> Higher levels of dirt and pollutants originating from, for example, combustion, tire and brake wear, and road surface degradation accumulate in road tunnels than in open road environments. Consequently, tunnel wash water is considerably more polluted than road runoff and is considered a hot spot for the emission of road-related

**Received:** November 10, 2023

**Revised:** December 13, 2023

**Accepted:** December 13, 2023

**Published:** December 22, 2023



pollutants.<sup>18–20</sup> These features also make tunnels ideal for studying emissions of road- and vehicle-related pollutants.

The objectives of this study were (1) to screen and assess the temporal concentrations of TWP and TDCs in tunnel wash water and (2) to assess the retention and treatment of TWP and TDCs in a tunnel wash water treatment basin.

For the first time, the concentrations of TWP and TDCs were investigated together during a whole tunnel wash event, including a 21-day long treatment period and the discharge of the treated tunnel wash water.

## MATERIALS AND METHODS

**Tunnel Description and Field Sampling.** The study was conducted from November 17 to December 8, 2022, in the 820 m long Vålereng road tunnel (Europe Road, E6) in Oslo, Norway (59.905465°N, 10.785367°E). The tunnel has two tubes, with three and two lanes in the northbound and southbound tubes, respectively. The annual average daily traffic (ADT) was 66 400 vehicles/day (10% heavy vehicles) in 2022. The driving speed is 60 km/h. The tunnel is typically washed 10–12 times per year. The tunnel wash water is treated in a sedimentation basin for 21 days before being discharged to a local stream. See the [Supporting Information](#) for more details.

A mixture of 39 m<sup>3</sup> of water and 50 L of soap [sodium dodecylbenzenesulfonate (CAS Registry No. 68411-30-3) and sodium lauryl ether sulfate (CAS Registry No. 9004-82-4)] was used during the tunnel cleaning of the southbound tunnel tube. In total, 33 water samples were collected for TWP analysis: 15 during the 1 h washing event (W1–W15), three during treatment [7, 14, and 21 days after washing (T16–T18, respectively)], and 15 during a period of 3.5 h of the discharge of treated wash water (D19–D33) (see [Figure S2](#)). All samples were collected using a metal sample collector (1 L) connected to a telescopic pole and taken approximately 35 cm below the surface. During samplings W1, W7, and W14 during washing and D19, D25, and D32 during discharge, samples were taken for TDC analysis. Such samples were also collected from the sedimentation basin on days 7, 14, and 21 (samples T16–T18, respectively).

Samples for TWP were collected in 1 L plastic bottles (HDPE), stored cold (4 °C) before being filtered on glass fiber filters (GF-A, 1.6 μm), and dried prior to analysis. Samples for TDCs were filtered through glass fiber filters (GF-A, 1.6 μm), conserved *in situ* on MeOH-activated HLB solid phase extraction cartridges (Waters), and frozen (–20 °C) prior to analysis. Different sampling volumes were collected due to clogging of the filters: W1 (70 mL), W7 and W14 (17.5 mL), and T16–T18 and D19 (35 mL).

**Chemical Analysis of TWPs.** The concentrations of TWPs were determined by pyrolysis gas chromatography mass spectrometry (PYR-GC/MS), following the methods by Rødland et al.<sup>21</sup> using the markers benzene (*m/z* 78), *α*-methylstyrene (*m/z* 117), ethylstyrene (*m/z* 118), and butadiene trimer (*m/z* 91). All samples were spiked with an internal standard [IS, deuterated polybutadiene d6-PB (*m/z* 60), Polymer Source, Inc., Dorval, QC], and the instrument performance was monitored using quality control (QC) samples during all sample runs [QC, 20 μg of styrene butadiene rubber (SBR1500, SBR+dPB, Polymer Source, Inc.)]. A description of the calibration and the QC performance can be found in the [Supporting Information](#) ([Figures S3 and S4](#) and [Table S4](#)). As styrene butadiene styrene (SBS) is added to the bitumen in the asphalt, it is

expected that rubbers from the road surface will be present in the samples. Identical pyrolysis products are produced from SBS and SBR, and the results from PYR-GC/MS will thus be a sum of SBR, BR, and SBS.<sup>22</sup> The expected ratio of SBR and BR from tires to SBS from PMB is applied to calculate the final rubber concentration from each source. The sum of SBR and BR is further used to predict the TWP concentration in the sample using reference tire tread and Monte Carlo simulation described by Rødland et al.<sup>22</sup>

**Chemicals and Reagents.** Reference standards of 6PPD, *N*-(5-methyl-2-hexyl)-*N*'-phenyl-*p*-phenylenediamine (7PPD), *N,N*'-bis(1,4-dimethylpentyl)-4-phenylenediamine (77PD), *N*-isopropyl-*N*'-phenyl-1,4-phenylenediamine (IPPD), *N*-cyclohexyl-*N*'-phenyl-*p*-phenylenediamine (CPPD), *N,N*'-diphenyl-1,4-phenylenediamine (DPPD), *N,N*'-di-2-naphthyl-*p*-phenylenediamine (DNP), 6PPD-Q, 2-aminobenzothiazole (ABT), 2-hydroxybenzothiazole (OHBT), 2-methylthiobenzothiazole (MBT), 2-phenylbenzothiazole (PhBT), benzothiazole-2-sulfonic (BTSA), 2,4,6-tris[bis(methoxymethyl)amino]-1,3,5-triazine (HMMM), 1,3-diphenylguanidine (DPG), and poly-(1,2-dihydro-2,2,4-trimethylquinoline) (TMQ) were obtained from TCI (Shang Hai, China), Accustandard (New Haven, CT), and Sigma-Aldrich (St. Louis, MO). Internal standards D14 DPPD and D5 6PPD-Q were purchased from Cambridge Isotope Laboratories Inc. (Tewksbury, MA) and Chiron AS (Trondheim, Norway). High-performance liquid chromatography (HPLC)-grade acetonitrile, methanol, and dichloromethane were purchased from Merck (Dusseldorf, Germany), and Milli-Q water was prepared using a Millipore pure water system. Solid phase extraction columns were Oasis HLB columns (200 mg, 6 cm<sup>3</sup>) from Waters (Milford, MA).

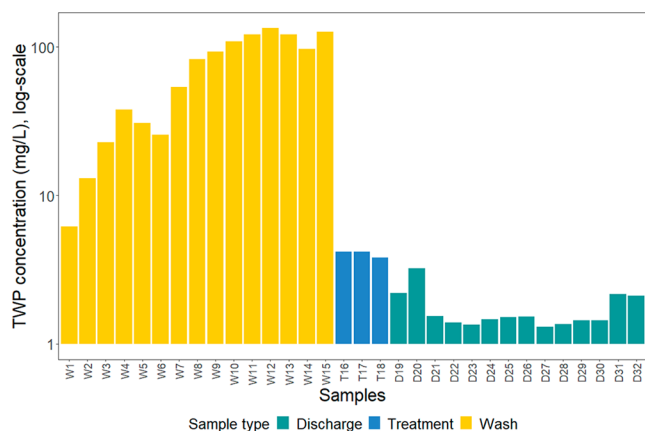
**Chemical Analysis of TDCs.** Samples were prepared and analyzed following the methods based on the work of Zhang et al.<sup>23</sup> and Huang et al.<sup>24</sup> with minor modifications. The HLB columns were thawed, and 100 ng of each IS was added. The analytes were eluted from the HLB cartridge with 8 mL of a 1:1:1 (v/v/v) MeOH/ACN/DCM mixture. The extract was concentrated to 2 mL under nitrogen. One milliliter of the extracts was centrifuged with a 0.2 μm nylon Spin-X filter (Costar, Salt Lake City, UT) for screening analysis, while 0.5 mL of the extracts was diluted with 0.5 mL of 5 mM aqueous ammonium formate and Spin-X filtered prior to quantitative analysis. Samples were further diluted to bring the concentrations of TDCs within the calibration range (1–200 ng/mL) as necessary. All reference standards and samples were analyzed by ultraperformance liquid chromatography time-of-flight mass spectrometry (UPLC-TOF-MS) HRMS for screening and identification of TDCs, while UPLC triple quadrupole mass spectrometry (UPLC-MSMS) was used for target quantitation of the TDCs of interest (see [Tables S1 and S2](#)).

**Quality Assurance and Quality Control.** Internal standards of DPPD and 6PPD-Q were added to the HLB column prior to elution/extraction to monitor the HLB extraction step and to control sample dilution; hence, the IS was not used to adjust any results. One laboratory procedural blank and two spiked water samples (tap water spiked with 100 ng of each reference standard) were processed along with the batch of samples. Recoveries of the target analytes in spiked water were in the range of 62–110%, and the relative standard deviations for the targeted chemicals were in the range of 2.9–6.3%. The instrumental LOD for the analytes was in the range of 0.2–0.5 ng/mL with a 2 μL injection volume on both

instruments. As the water samples contained high levels of particulates and soap, only a small volume of the water samples could be processed (typically 7–70 mL), thus giving a relatively high method LOD ranging from 15 to 60 ng/L. For more information about the analytical performance, see Table S3.

## RESULTS AND DISCUSSION

**TWP Concentrations.** The concentrations of TWPs ( $>1.6 \mu\text{m}$ ) increased from the beginning to the end (Figure 1 and



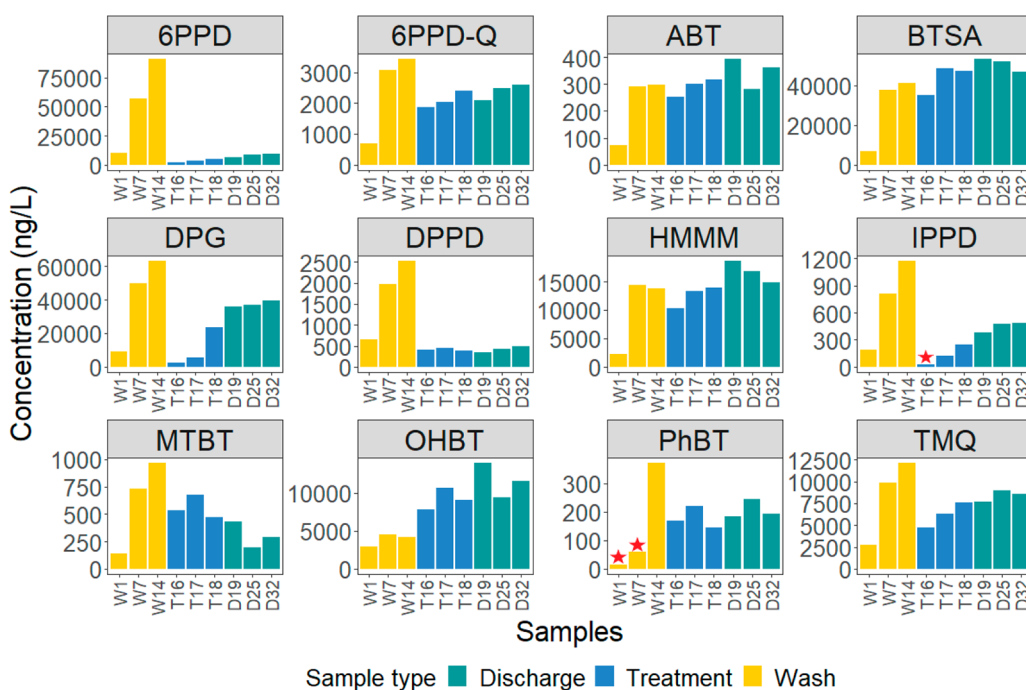
**Figure 1.** Bar plot showing the tire wear particle (TWP) concentrations measured during the tunnel wash event (yellow,  $\sim 1$  h), during treatment (blue, days 7, 14, and 21), and during the discharge of treated tunnel wash water (green,  $\sim 3.5$  h). The y-axis is log-scaled to better show the variation in TWP concentrations. All data can be found in Table S5.

Table S5), but with a slight bimodal pattern. The peak concentration was observed for sample W12,  $134 \pm 39.7$  mg/L

(mean  $\pm$  standard deviation), and the mean and median concentrations were 71.8 and 67.5 mg/L, respectively. The TWP concentrations were substantially higher than those measured in the nearby Smestad tunnel (tunnel wash, 14.5–47.8 mg/L TWP; discharge, 6.78–29.4 mg/L TWP).<sup>22</sup> This discrepancy may be attributed to higher traffic levels in the Vålereng tunnel than in the Smestad tunnel [ADT of 66 400 vs ADT of 44 000 (both tubes)]. In comparison, the TWP concentration in the Vålereng tunnel was substantially lower than the TWP concentrations found in road-side snow in Oslo (76–14 500 mg/L TWP).<sup>4</sup>

**Identification and Quantification of TDCs.** The high-resolution UPLC-MS method was first applied for screening the chemical profiles of the collected tunnel wash samples. All of the available reference standards were also analyzed to obtain data on their chromatographic behavior as well as their ionization and fragmentation processes. On the basis of the findings, an UPLC-MSMS target quantification method was set up to quantify the TDCs of interest (Table S2).

**TDC Concentrations.** The results represent the TDCs detected in the  $<1.6 \mu\text{m}$  fraction of the water samples, thus containing both the leached chemicals and a proportion of small particles and colloids in suspension. Six of the 18 TDCs were not detected in any of the samples [CPPD, DNPD, 7PPD, 44PD, 77PD, and MBT (Table S6)]. Despite the less frequent sampling of TDCs, their concentration pattern during the tunnel wash event aligned with the more frequent TWP sampling. In general, the lowest concentrations were found at the beginning of the washing while the highest concentrations were typically observed in the last sampling. This suggests a close relationship between the observed TDC concentrations and the emission of TWPs despite them being measured in different size fractions (Figure 2).



**Figure 2.** Bar plot showing the tire-derived chemicals (TDCs) measured during the tunnel wash event (blue,  $\sim 1$  h), during treatment (green, days 7, 14, and 21), and during the discharge of treated tunnel wash water (yellow,  $\sim 3.5$  h). Values below the LOD are displayed but indicated by a red star. All data can be found in Table S6.

The concentrations of 6PPD and 6PPD-Q peaked at the third washing event sample at 91 200 and 3440 ng/L, respectively. Our study reported higher concentrations of 6PPD-Q compared to those found in urban runoff from Hong Kong (210–2430 ng/L)<sup>25</sup> and road runoff from Huizhou and Dongguan (38.5–1562 ng/L).<sup>23</sup> The concentrations of 6PPD were much higher than those reported elsewhere, e.g., 300–14 300 ng/L in a German wastewater treatment plant<sup>26</sup> and 21–130 ng/L in road runoff from Pierce County (USA).<sup>27</sup> According to Zhou et al.,<sup>28</sup> the presence of organic matter and sunlight may enhance the formation of 6PPD-Q in a continuous manner. Hence, the high levels of 6PPD in the discharged tunnel wash water are concerning.

Several other TDCs (ABT, HMMM, DPG, OHBT, and MTBT) measured in the tunnel wash water showed in most cases considerably higher concentrations compared to those in surface water during storm events previously reported.<sup>7,23,29</sup> In road runoff, HMMM concentrations ranged between 2800 and 9100 ng/L,<sup>30</sup> which is approximately half of the HMMM peak concentration in our study (18 629 ng/L). Challis et al.<sup>31</sup> reported exceptionally high concentrations of DPG in stormwater from Saskatoon (Canada), peaking at >300 000 ng/L, while Peter et al.<sup>30</sup> estimated DPG concentrations to be ~1800 ng/L in Seattle. The peak concentration of DPG in our study was 63 086 ng/L, fairly similar to that of road runoff from Huizhou and Dongguan (58 780 ng/L).<sup>23</sup> Zhang et al.<sup>23</sup> also measured DPPD, IPPD, CPPD, DNPd, TMQ, OHBT, ABT, and MTBT, where only the concentrations of ABT and MTBT were higher than those in the tunnel wash water (69.9–13 260 and 180–4170 ng/L, respectively). OHBT concentrations were comparable (941–9292 ng/L) to those of the tunnel wash water, while DPPD (0.06–0.59 ng/L) and IPPD (0.45–1.93 ng/L) concentrations were considerably lower than those in the tunnel wash water. The tunnel wash water is retained in a closed sedimentation basin that will prevent photolysis that otherwise may be a pathway of transformation of tire additives in the natural environment. This may be an explanation of why we measured higher concentrations in our study. Additionally, anoxic conditions, which are common in tunnel treatment basins, may also influence the transformation of 6PPD to 6PPD-Q as shown in anaerobic flooded soils.<sup>32</sup> Finally, care should be taken when comparing data between different studies. For example, we used a 1.6  $\mu\text{m}$  filter cutoff, while Zhang et al.<sup>23</sup> used a 1.2  $\mu\text{m}$  filter cutoff. Hence, our comparisons should be interpreted as an indication of differences rather than exact differences.

Overall, the concentrations of TDCs measured in the tunnel washwater appear to be equal to or higher than previous measurements, highlighting that tunnels are significant hot spots for the emission of pollutants into the aquatic environment.

**Treatment Performance.** The removal of TDCs in tunnel wash water reached 98% after treatment for 21 days, demonstrating that sedimentation is an effective method for treating such particles (Table S7). However, the nearby Smestad tunnel showed less efficient treatment performance (63%),<sup>22</sup> which may be attributed to the design of the water pumping solution in and out of the sedimentation basin rather than the sedimentation process. Initial observations indicated sludge resuspension during the discharge of treated tunnel wash water in the Smestad tunnel.

The treatment performance of TDCs was calculated for sedimentation treatment for 7, 14, and 21 days, as well as for

the discharge treated water on day 21 (Table S7). The highest treatment performances for 6PPD (96%), DPG (93%), TMQ (42%), and 6PPD-Q (22%) were observed on day 7. Two compounds exhibited higher treatment performance on day 21 [PhBT (61%) and MTBT (50%)], while IPPD had its highest treatment on day 14 (84%). On day 21, no treatment (0.5%) of 6PPD-Q was evident, and several compounds displayed negative treatment performance with higher concentrations in the discharged water than in the inlet water. The highest negative treatment performance was observed for OHBT (−199%), followed by BTSA (−79%), ABT (−58%), and TMQ (−2%). There was a strong positive relationship between the TDCs' treatment performance (ranked) and their octanol/water partition coefficient ( $\log K_{ow}$ ) ( $R^2 = 0.71$ ;  $p = 0.006$ ) (Figure S5). These results indicate that several of the measured TDCs are poorly retained in the sedimentation basin and may end up in the environment due to their leachability and mobility. We believe that the soap may have a strong influence on the fate and behavior of the TDCs in the treatment system by, e.g., affecting their desorption and solubilization from particles, micelle formation encapsulating the TDCs, and influencing colloidal stability. Xia et al.<sup>33</sup> showed, for example, that soaps may even have a substantial impact on how microplastic particles adsorb ionic organic pollutants. Hence, the implications the soap may have on the tunnel wash treatment process and the fate of TDCs warrant further study.

**Environmental Implications.** This study revealed high concentrations of several TDCs in both untreated and treated tunnel wash water. This raises concerns as TDCs may pose a toxic risk to aquatic organisms.<sup>25,34</sup> Additionally, most tunnels lack washwater treatment or rely solely on sedimentation basins. Many countries worldwide utilize sedimentation basins or nature-based solutions like wetlands, retention, and detention ponds for treating road runoff and urban stormwater. Therefore, our findings regarding tunnel wash water may be applicable to such conditions. However, the retention and treatment of TDCs in nature-based solutions may exhibit different patterns. Factors such as biochemical and photochemical processes, temperature fluctuations, seasonality, uptake in plants, and other abiotic and biotic factors are likely to influence treatment performance. Future studies should explore the fate and effective retention of TDCs in these systems.

## ■ ASSOCIATED CONTENT

### SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.estlett.3c00811>.

Details about the treatment basin and sampling locations, sampling scheme, instrumental analysis, including quality control and quality assurance with UPLC-TOF-MS, UPLC-MSMS, and PYR-GC/MS, results of tire rubber concentrations and estimated TWP concentrations, TDC concentrations, and treatment performance of TDCs (PDF)

## ■ AUTHOR INFORMATION

### Corresponding Author

Sondre Meland – Norwegian Institute for Water Research, NO-0579 Oslo, Norway; [orcid.org/0000-0002-7497-4209](https://orcid.org/0000-0002-7497-4209); Email: [sondre.meland@niva.no](mailto:sondre.meland@niva.no)

## Authors

**Gina Marie Granheim** – Norwegian University of Life Sciences, Faculty of Environmental Sciences and Natural Resource Management, NO-1432 Ås, Norway

**Jan Thomas Rundberget** – Norwegian Institute for Water Research, NO-0579 Oslo, Norway

**Elisabeth Rødland** – Norwegian Institute for Water Research, NO-0579 Oslo, Norway; [orcid.org/0000-0002-2060-6101](https://orcid.org/0000-0002-2060-6101)

Complete contact information is available at:

<https://pubs.acs.org/10.1021/acs.estlett.3c00811>

## Notes

The authors declare no competing financial interest.

## ACKNOWLEDGMENTS

This work was funded by the Norwegian Public Roads Administration (Grant O-210147) and the Research Council of Norway (Grant 160016).

## REFERENCES

- (1) Kole, P. J.; Lohr, A. J.; Van Belleghem, F. G. A. J.; Ragas, A. M. J. Wear and Tear of Tyres: A Stealthy Source of Microplastics in the Environment. *Int. J. Environ. Res. Public Health* **2017**, *14* (10), 1265.
- (2) Unice, K. M.; Weeber, M. P.; Abramson, M. M.; Reid, R. C. D.; van Gils, J. A. G.; Markus, A. A.; Vethaak, A. D.; Panko, J. M. Characterizing Export of Land-Based Microplastics to the Estuary - Part I: Application of Integrated Geospatial Microplastic Transport Models to Assess Tire and Road Wear Particles in the Seine Watershed. *Sci. Total Environ.* **2019**, *646*, 1639–1649.
- (3) Fussell, J. C.; Franklin, M.; Green, D. C.; Gustafsson, M.; Harrison, R. M.; Hicks, W.; Kelly, F. J.; Kishita, F.; Miller, M. R.; Mudway, I. S.; Oroumijeh, F.; Selley, L.; Wang, M.; Zhu, Y. A Review of Road Traffic-Derived Non-Exhaust Particles: Emissions, Physicochemical Characteristics, Health Risks, and Mitigation Measures. *Environ. Sci. Technol.* **2022**, *56* (11), 6813–6835.
- (4) Rødland, E. S.; Lind, O. C.; Reid, M. J.; Heier, L. S.; Okoffo, E. D.; Rauert, C.; Thomas, K. V.; Meland, S. Occurrence of Tire and Road Wear Particles in Urban and Peri-Urban Snowbanks, and Their Potential Environmental Implications. *Sci. Total Environ.* **2022**, *824*, No. 153785.
- (5) Rodland, E. S.; Gustafsson, M.; Jaramillo-Vogel, D.; Järnskog, I.; Müller, K.; Rauert, C.; Rausch, J.; Wagner, S. Analytical Challenges and Possibilities for the Quantification of Tire-Road Wear Particles. *TrAC, Trends Anal. Chem.* **2023**, *165*, No. 117121.
- (6) Kreider, M. L.; Unice, K. M.; Panko, J. M. Human Health Risk Assessment of Tire and Road Wear Particles (TRWP) in Air. *Hum. Ecol. Risk Assess.* **2020**, *26* (10), 2567–2585.
- (7) Rauert, C.; Charlton, N.; Okoffo, E. D.; Stanton, R. S.; Agua, A. R.; Pirrung, M. C.; Thomas, K. V. Concentrations of Tire Additive Chemicals and Tire Road Wear Particles in an Australian Urban Tributary. *Environ. Sci. Technol.* **2022**, *56* (4), 2421–2431.
- (8) Johannessen, C.; Helm, P.; Metcalfe, C. D. Detection of Selected Tire Wear Compounds in Urban Receiving Waters. *Environ. Pollut.* **2021**, *287*, No. 117659.
- (9) Wagner, S.; Huffer, T.; Klockner, P.; Wehrhahn, M.; Hofmann, T.; Reemtsma, T. Tire Wear Particles in the Aquatic Environment - A Review on Generation, Analysis, Occurrence, Fate and Effects. *Water Res.* **2018**, *139*, 83–100.
- (10) Baensch-Baltruschat, B.; Kocher, B.; Stock, F.; Reifferscheid, G. Tyre and Road Wear Particles (TRWP) - A Review of Generation, Properties, Emissions, Human Health Risk, Ecotoxicity, and Fate in the Environment. *Sci. Total Environ.* **2020**, *733*, No. 137823.
- (11) USTMA. What's in a tire. <https://www.ustires.org/whats-tire-0> (accessed 2023-12-12).
- (12) Müller, K.; Hübner, D.; Huppertsberg, S.; Knepper, T. P.; Zahn, D. Probing the Chemical Complexity of Tyres: Identification of Potential Tire-Borne Water Contaminants with High-Resolution Mass Spectrometry. *Sci. Total Environ.* **2022**, *802*, No. 149799.
- (13) Tian, Z.; Zhao, H.; Peter, K. T.; Gonzalez, M.; Wetzel, J.; Wu, C.; Hu, X.; Prat, J.; Mudrock, E.; Hettlinger, R.; Cortina, A. E.; Biswas, R. G.; Kock, F. V. C.; Soong, R.; Jenne, A.; Du, B.; Hou, F.; He, H.; Lundeen, R.; Gilbreath, A.; Sutton, R.; Scholz, N. L.; Davis, J. W.; Dodd, M. C.; Simpson, A.; McIntyre, J. K.; Kolodziej, E. P. A Ubiquitous Tire Rubber-Derived Chemical Induces Acute Mortality in Coho Salmon. *Science* **2021**, *371*, No. 185.
- (14) Markiewicz, A.; Bjorklund, K.; Eriksson, E.; Kalmykova, Y.; Stromvall, A. M.; Siopi, A. Emissions of Organic Pollutants from Traffic and Roads: Priority Pollutants Selection and Substance Flow Analysis. *Sci. Total Environ.* **2017**, *580*, 1162–1174.
- (15) Muller, A.; Osterlund, H.; Marsalek, J.; Viklander, M. The Pollution Conveyed by Urban Runoff: A Review of Sources. *Sci. Total Environ.* **2020**, *709*, 136125.
- (16) Meland, S.; Gomes, T.; Petersen, K.; Häll, J.; Lund, E.; Kringstad, A.; Grung, M. Road Related Pollutants Induced DNA Damage in Dragonfly Nymphs (Odonata, Anisoptera) Living in Highway Sedimentation Ponds. *Sci. Rep.* **2019**, *9* (1), 16002.
- (17) Meland, S.; Borgstrom, R.; Heier, L. S.; Rosseland, B. O.; Lindholm, O.; Salbu, B. Chemical and Ecological Effects of Contaminated Tunnel Wash Water Runoff to a Small Norwegian Stream. *Sci. Total Environ.* **2010**, *408* (19), 4107–4117.
- (18) Meland, S. Management of Contaminated Runoff Water. Current Practice and Future Research Needs. CEDR report; 2016/01; Conference of European Directors of Roads (CEDR): Brussels, 2016; p 84 (<https://www.cedr.eu/download/Publications/2016/CEDR2016-1-Management-of-contaminated-runoff-water.pdf>).
- (19) Hallberg, M.; Renman, G.; Byman, L.; Svenstam, G.; Norling, M. Treatment of Tunnel Wash Water and Implications for Its Disposal. *Water Sci. Technol.* **2014**, *69* (10), 2029–2035.
- (20) Meland, S.; Heier, L. S.; Salbu, B.; Tollefsen, K. E.; Farmen, E.; Rosseland, B. O. Exposure of Brown Trout (*Salmo Trutta* L.) to Tunnel Wash Water Runoff - Chemical Characterisation and Biological Impact. *Sci. Total Environ.* **2010**, *408* (13), 2646–2656.
- (21) Rødland, E. S.; Samanipour, S.; Rauert, C.; Okoffo, E. D.; Reid, M. J.; Heier, L. S.; Lind, O. C.; Thomas, K. V.; Meland, S. A Novel Method for the Quantification of Tire and Polymer-Modified Bitumen Particles in Environmental Samples by Pyrolysis Gas Chromatography Mass Spectroscopy. *J. Hazard. Mater.* **2022**, *423*, No. 127092.
- (22) Rodland, E. S.; Lind, O. C.; Reid, M.; Heier, L. S.; Skogsberg, E.; Snilsberg, B.; Gryteselv, D.; Meland, S. Characterization of Tire and Road Wear Microplastic Particle Contamination in a Road Tunnel: From Surface to Release. *J. Hazard. Mater.* **2022**, *435*, No. 129032.
- (23) Zhang, H. Y.; Huang, Z.; Liu, Y. H.; Hu, L. X.; He, L. Y.; Liu, Y. S.; Zhao, J. L.; Ying, G. G. Occurrence and Risks of 23 Tire Additives and Their Transformation Products in an Urban Water System. *Environ. Int.* **2023**, *171*, 107715.
- (24) Huang, W.; Shi, Y.; Huang, J.; Deng, C.; Tang, S.; Liu, X.; Chen, D. Occurrence of Substituted P-Phenylenediamine Antioxidants in Dusts. *Environ. Sci. Technol. Lett.* **2021**, *8* (5), 381–385.
- (25) Chen, X.; He, T.; Yang, X.; Gan, Y.; Qing, X.; Wang, J.; Huang, Y. Analysis, Environmental Occurrence, Fate and Potential Toxicity of Tire Wear Compounds 6PPD and 6PPD-Quinone. *J. Hazard. Mater.* **2023**, *452*, No. 131245.
- (26) Zoroufchi Benis, K.; Behnami, A.; Minaei, S.; Brinkmann, M.; McPhedran, K. N.; Soltan, J. Environmental Occurrence and Toxicity of 6PPD Quinone, an Emerging Tire Rubber-Derived Chemical: A Review. *Environ. Sci. Technol. Lett.* **2023**, *10* (10), 815–823.
- (27) Zhao, H. N.; Hu, X.; Tian, Z.; Gonzalez, M.; Rideout, C. A.; Peter, K. T.; Dodd, M. C.; Kolodziej, E. P. Transformation Products of Tire Rubber Antioxidant 6PPD in Heterogeneous Gas-Phase Ozonation: Identification and Environmental Occurrence. *Environ. Sci. Technol.* **2023**, *57* (14), 5621–5632.

(28) Zhou, Y.; Yixi, L.; Kong, Q.; Peng, J.; Pan, Y.; Qiu, J.; Yang, X. Sunlight-Induced Transformation of Tire Rubber Antioxidant N-(1,3-Dimethylbutyl)-N'-Phenyl-p-Phenylenediamine (6PPD) to 6PPD-Quinone in Water. *Environ. Sci. Technol. Lett.* **2023**, *10* (9), 798–803.

(29) Johannessen, C.; Helm, P.; Metcalfe, C. D. Runoff of the Tire-Wear Compound, Hexamethoxymethyl-Melamine into Urban Watersheds. *Arch. Environ. Contam. Toxicol.* **2022**, *82* (2), 162–170.

(30) Peter, K. T.; Tian, Z.; Wu, C.; Lin, P.; White, S.; Du, B.; McIntyre, J. K.; Scholz, N. L.; Kolodziej, E. P. Using High-Resolution Mass Spectrometry to Identify Organic Contaminants Linked to Urban Stormwater Mortality Syndrome in Coho Salmon. *Environ. Sci. Technol.* **2018**, *52* (18), 10317–10327.

(31) Challis, J. K.; Popick, H.; Prajapati, S.; Harder, P.; Giesy, J. P.; McPhedran, K.; Brinkmann, M. Occurrences of Tire Rubber-Derived Contaminants in Cold-Climate Urban Runoff. *Environ. Sci. Technol. Lett.* **2021**, *8* (11), 961–967.

(32) Xu, Q.; Li, G.; Fang, L.; Sun, Q.; Han, R.; Zhu, Z.; Zhu, Y.-G. Enhanced Formation of 6PPD-Q during the Aging of Tire Wear Particles in Anaerobic Flooded Soils: The Role of Iron Reduction and Environmentally Persistent Free Radicals. *Environ. Sci. Technol.* **2023**, *57* (14), 5978–5987.

(33) Xia, Y.; Zhou, J.-J.; Gong, Y.-Y.; Li, Z.-J.; Zeng, E. Y. Strong Influence of Surfactants on Virgin Hydrophobic Microplastics Adsorbing Ionic Organic Pollutants. *Environ. Pollut.* **2020**, *265*, No. 115061.

(34) Zhang, R. L.; Zhao, S. Z.; Liu, X.; Tian, L. L.; Mo, Y. Z.; Yi, X.; Liu, S. Y.; Liu, J. Q.; Li, J.; Zhang, G. Aquatic Environmental Fates and Risks of Benzotriazoles, Benzothiazoles, and p-Phenylenediamines in a Catchment Providing Water to a Megacity of China. *Environ. Res.* **2023**, *216*, 114721.