



# Toxic Tire Wear Compounds (6PPD-Q and 4-ADPA) Detected in Airborne Particulate Matter Along a Highway in Mississippi, USA

Boluwatife S. Olubusoye<sup>1</sup> · James V. Cizdziel<sup>1</sup> · Matthew Bee<sup>1</sup> · Matthew T. Moore<sup>2</sup> · Marco Pineda<sup>3</sup> · Viviane Yargeau<sup>3</sup> · Erin R. Bennett<sup>4</sup>

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## Abstract

Tire wear particles (TWPs) are a major category of microplastic pollution produced by friction between tires and road surfaces. This non-exhaust particulate matter (PM) containing leachable toxic compounds is transported through the air and with stormwater runoff, leading to environmental pollution and human health concerns. In the present study, we collected airborne PM at varying distances (5, 15 and 30 m) along US Highway 278 in Oxford, Mississippi, USA, for ten consecutive days using Sigma-2 passive samplers. Particles (~ 1–80 µm) were passively collected directly into small (60 mL) wide-mouth separatory funnels placed inside the samplers. Particles were subsequently subjected to solvent extraction, and extracts were analyzed for TWP compounds by high resolution orbitrap mass spectrometry. This pilot study was focused solely on qualitative analyses to determine whether TWP compounds were present in this fraction of airborne PM. The abundance of airborne TWPs increased with proximity to the road with deposition rates (TWPs cm<sup>-2</sup> day<sup>-1</sup>) of 23, 47, and 63 at 30 m, 15 m, and 5 m from the highway, respectively. Two common TWP compounds (6PPD-Q and 4-ADPA) were detected in all samples, except the field blank, at levels above their limits of detection, estimated at 2.90 and 1.14 ng L<sup>-1</sup>, respectively. Overall, this work suggests airborne TWPs may be a potential inhalation hazard, particularly for individuals and wildlife who spend extended periods outdoors along busy roadways. Research on the bioavailability of TWP compounds from inhaled TWPs is needed to address exposure risk.

**Keywords** Tire wear particles · Tire wear compounds · 6PPD-Q · 4-ADPA · Sigma-2 passive sampler · Airborne particulate matter · Road traffic

Tire wear particles (TWPs) are of environmental concern because they can release chemicals, heavy metals, and other additives that have adverse effects on aquatic and terrestrial organisms, and because the amount of TWPs released may rival all other microplastics combined (Sundt et al. 2014; Wagner et al. 2018; Zahn et al. 2019; Seiwert

et al. 2020; Masset et al. 2022; Chen et al. 2023). The tire wear compound N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) and its oxidative transformation product 6PPD-quinone (6PPD-Q) are considered emerging pollutants, having recently been measured in road runoff from urban areas (Johannessen et al. 2022) and shown to induce acute mortality in coho salmon (*Oncorhynchus kisutch*) and other fish (Tian et al. 2020; Brinkmann et al. 2022). Further, Halle et al. (2021) conducted a study demonstrating the potential impact of TWPs on both the acute mortality and long-term growth of the crustacean *Hyaella azteca* in aquatic environments, while Fang et al. (2023) reported bioaccumulation of 6PPD and 6PPD-Q on the liver of mice, especially at a dose of 100 mg kg<sup>-1</sup>. Additionally, hepatotoxicity in mice from 6PPD and 6PPD-Q were reported at 100 mg kg<sup>-1</sup> concentrations of both 6PPD and 6PPD-Q (Fang et al. 2023).

✉ James V. Cizdziel  
cizdziel@olemiss.edu

<sup>1</sup> Department of Chemistry and Biochemistry, University of Mississippi, Oxford, MS 38677, USA

<sup>2</sup> Water Quality and Ecology Research Unit, Agricultural Research Service, U.S. Department of Agriculture, Oxford, MS 38655, USA

<sup>3</sup> Department of Chemical Engineering, McGill University, Montreal, QC, Canada

<sup>4</sup> School of the Environment, Trent University, Peterborough, ON, Canada

TWPs are characterized as dark/black particles often elongated or cylindrical in shape that can range in size from ~ 1 to > 100  $\mu\text{m}$  (Kreider et al. 2010; Sommer et al. 2018). Friction and other mechanical forces between tires and the road generate fragments of tire tread encrusted with pavement and road related particles (Panko et al. 2018; Baensch-Baltruschat et al. 2020; Rausch et al. 2022). Incorporation of road particles alters the chemical composition of TWPs from that of pure tire tread and tire wear debris (Kreider et al. 2010). Baensch-Baltruschat et al. (2020) reviewed TWP occurrence, transport, fate, and ecological and human health risk. They identified several key areas of research which need further exploration including determination of more precise emission factors for different types of roads and surfaces; monitoring and field studies to determine actual environmental concentrations and degradation of materials under realistic environmental conditions; ecotoxicology assessments with marine and freshwater species using realistic concentrations; and model development to estimate fate of TWP in aquatic and terrestrial environments (Baensch-Baltruschat et al. 2020).

Inhalation of airborne PM can result in deleterious human health effects (e.g., Gordon et al. 2012; Brunekreef and Forsberg 2005; Gerking et al. 2014), and deposition of PM to terrestrial and aquatic ecosystems can also have serious impacts (Gordon et al. 2012; Mirowsky et al. 2013). For TWPs specifically, inhaled TWPs have induced pulmonary fibrotic injury via epithelial cytoskeleton rearrangement in mice (Li et al. 2022). Environmentally persistent free radicals associated with photo-aged TWPs decreased cell viability, the increase of oxidative stress response, and inflammatory factor secretion using macrophages as model cells for bioassays (Liu et al. 2022). Thus, it is important to not only quantify and characterize airborne PM which contributes to air and water pollution, but also measure compounds that can leach from them.

To that end, we used Sigma-2 passive samplers to collect airborne PM (~ 1–80  $\mu\text{m}$ ) at varying distances (5, 15, and 30 m) from a common thoroughfare, US Highway 278, in Oxford, Mississippi, USA. The Sigma-2 passive sampler is a vertical flux sampler that been used primarily in Europe for air quality measurements, including TWPs, trace elements, isotope measurements, and PM source apportionment (e.g., Gueguen et al. 2012; Tian et al. 2017; Sommer et al. 2018; Rausch et al. 2022). We have previously used Sigma-2 passive samplers to study the abundance and characteristics of airborne PM, including TWPs, along roads with different traffic characteristics (Gao et al. 2022). Samples were analyzed by single-particle scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) with machine learning to classify particles based on morpho-textual-chemical parameters into tire wear, metallic, mineral, and biogenic/organic particles (Sommer et al.

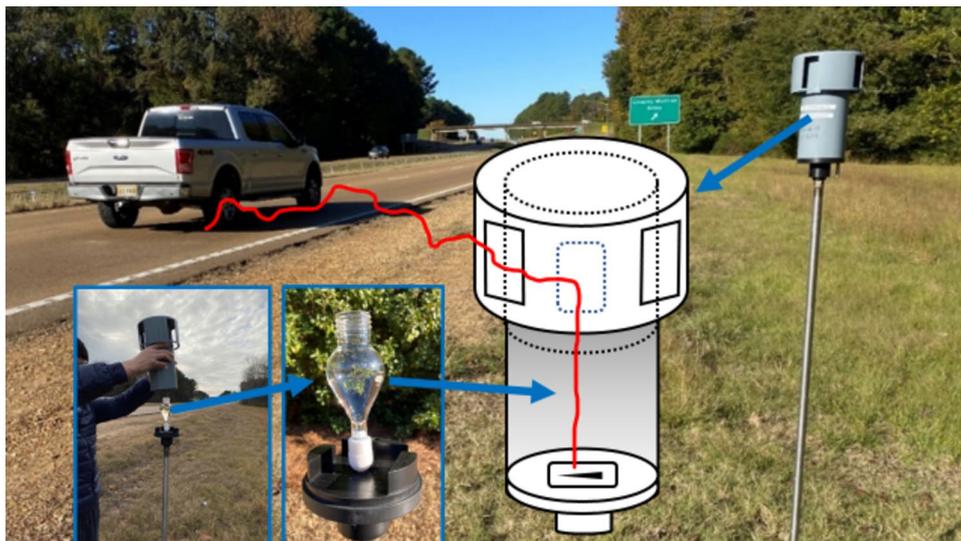
2018; Kovochich et al. 2021a, b; Rausch et al. 2022). Factors found to affect TWP abundance include proximity to the road, vehicle speed, traffic volume, and driving behavior (Gao et al. 2022).

In the present study, we modified the sampler to collect particles directly into small (60 mL) separatory funnels over a period of 10 days. Subsequently, we added solvents to extract TWP compounds from particles and analyzed extracts by high resolution orbitrap mass spectrometry. The aim being to determine if TWP marker compounds [e.g., 6-PPDQ and 4-aminodiphenylamine (4-ADPA)], could be detected in samples. Thus, this paper focuses solely on qualitative measurements to assess whether additional study is warranted; it does not include quantitative (concentration) measurements. Whereas previous air studies have detected atmospheric TWP (6-PPDs and 6-PPDQ) collected on quartz fiber filters in high volume samplers deployed in urban centers (Zhang et al. 2021; Cao et al. 2022), this is the first time TWP compounds have been detected in airborne PM collected passively along an active US highway. The advantages of passive sampling include low cost, no power, and greater potential for collecting spatially-resolved data, while sampling directly into the extraction vessel lowers the potential for contamination as well as negating any losses from transferring particles.

## Materials and Methods

Airborne PM was collected using Sigma-2 passive samplers (Particle Vision Ltd., Fribourg, Switzerland), which have been described elsewhere (VDI2119: 2011; Dietze et al. 2010; Tian et al. 2017). Briefly, air PM ranging from ~ 1 to ~ 80  $\mu\text{m}$  (geometric diameter) enters the sampler and, in the low-turbulent air within the sedimentation tube, deposits on substrates for analyses. The Sigma-2 design protects particles from direct sunlight, wind, and precipitation. Sigma-2 samplers were deployed 1.5 m above the ground using a stainless-steel rod but were modified by inserting a 60 mL Squibb glass separatory funnel with a PTFE valve to capture a subset of particles entering the samplers (Fig. 1). To add the funnel, a hole was drilled in the center of the Sigma-2 base plate allowing the funnel stem to be inserted to ensure it was securely upright. The top opening diameter of the separatory funnel was 2.5 cm, providing an area of 4.91  $\text{cm}^2$  for particles to fall. We have previously used the setup to facilitate density separations particle analyses since a high particle load can result in overlapping particles that complicate both particle counting and characterization (Gao et al. 2022). A density separation helps isolate microplastics from denser mineral or road particles prior to analyses. In this case, particles were collected directly into the separatory funnel for extraction and analyses. A field blank consisting

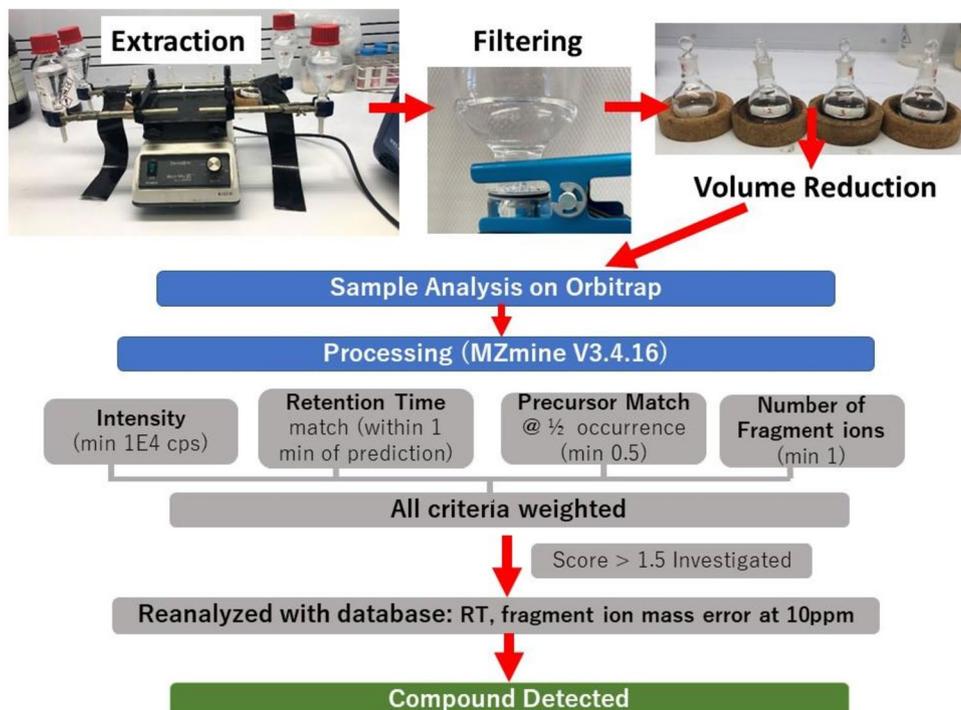
**Fig. 1** Sigma 2 sampler deployed along US Highway 278 in Oxford, Mississippi. The graphical image depicts a hypothetical path (red line) of an airborne TWP as it travels from the road through the air into the sampler and ultimately depositing on a sticky substrate (slide). In the present study, the slide was replaced with a 60 mL separatory funnel (blue boxes) to capture the particles as described in the text (Colo figure online)



of a separatory funnel briefly opened at the start of sampling but then closed for the duration of sampling was also collected. Sigma-2 samplers were deployed at three distances (5, 15, and 30 m) from US Highway 278 near the University of Mississippi (UM) campus located in Oxford, Mississippi, USA. The highway averages ~35,000 vehicles daily traveling at an average speed of ~105 km h<sup>-1</sup>. An estimated deposition rate of ~700 TWP cm<sup>-2</sup> day<sup>-1</sup> along the highway was reported (Gao et al. 2022). After a 10-day collection period, each separatory funnel was capped and transported to the laboratory.

The PM was extracted using an adapted method described by Fohet et al. (2023). The overall approach is shown in Fig. 2. Briefly, after separatory funnels were retrieved from sampling sites, 50 mL of a 50:50 mixture of methanol and hexane were added. Funnels were then covered with aluminum foil to minimize potential light-induced sample transformations. Separatory funnels were then placed on a shaker for 24 h. Subsequently, each solution was vacuum filtered (0.2 μm) using polycarbonate gold-coated filters (Steritech Corp.), and the filtrate was transferred into a round-bottom flask. Each sample was rotary evaporated to a volume of

**Fig. 2** General sample preparation and analyses scheme



approximately 3 mL. Samples were then shipped to McGill University for analysis by orbitrap mass spectrometry. For future work involving quantitation a surrogate should be added prior to extraction and processing to assess extraction efficiency and help monitor for matrix effects and gross sample processing errors.

Extracts from the PM were further reduced to 50  $\mu\text{L}$  with a nitrogen evaporator and resuspended with 200  $\mu\text{L}$  of a buffer (methanol–water at 2:3) and transferred to a conical glass vial for injection. Analysis of the TWP extracts was performed on a Vanquish UHPLC pump system coupled to a TriPlus RSH EQuan 850 autosampler from Thermo Scientific (Waltham, US) using a mass spectrometer Exploris 120 HRMS detector (Thermo Scientific, Bremen, Germany). Positive and negative mode in heated electrospray ionization mode (HESI) was used for the generation of precursor ions. Optimization of the mass spectrometry parameters was done by direct injection of a mix standard solution (0.5  $\text{mg mL}^{-1}$  containing 4-ADPA, 6PPD, 4-HDPA, 6PPD-Q, and NO-DPA) at 10  $\text{mL min}^{-1}$  and 1:1 solvent mix water to methanol at 250  $\text{mL min}^{-1}$  with the following parameters: positive and negative source capillary voltage at 3500 and 2500 V respectively; source and HCD collision gas was nitrogen on static mode at arbitrary gas units at 45, 21 and 2 for the sheath, auxiliary and sweep gasses, respectively. The ion transfer tube temperature used was 280°C, while the vaporization temperature applied during ionization was 300°C. Captured ions were focused on a beam with a 60% lens frequency. The acquisition was conducted in full scan mode (70–1000  $m/z$ ) with a FTMS resolution of 60,000 at 200  $m/z$ . Compound identification was performed by data-dependent MS2 scans at 15,000 FWHM using 6s dynamic exclusion and, the mass tolerance set at 5  $\text{mg L}^{-1}$  to generate the respective data-dependent product ion spectra at 2  $m/z$  isolation window and 1  $\mu$  scan with dd-MS2 scans set in normalized HCD collision energy at 15%, 30%, and 45%.

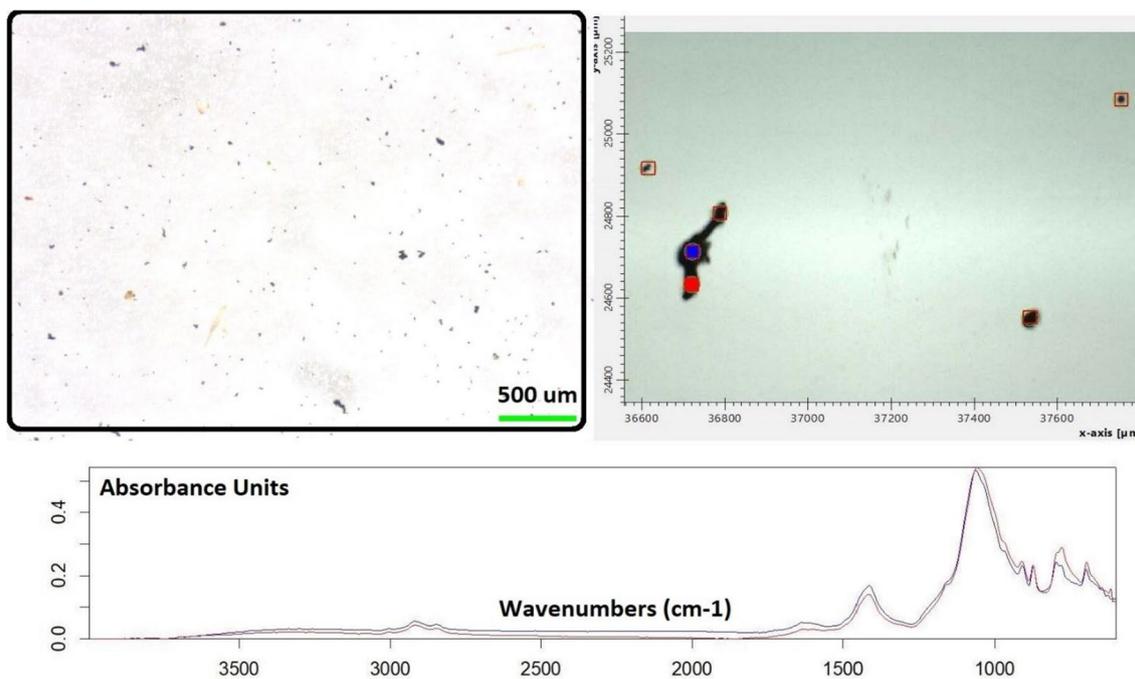
Analytical separation of samples was performed on a column assembly consisting of an online filter cartridge with a 2.1 mm ID  $\times$  0.2  $\mu\text{m}$  porosity stainless steel filter, an Eclipse Plus C18 RRHD (5 mm  $\times$  2.1 mm ID; 1.8  $\mu\text{m}$ ) guard column followed by the analytical column with the same stationary phase (100 mm  $\times$  2.1 mm, 1.8  $\mu\text{m}$ ) (Agilent, California, USA). Column temperature was kept constant throughout the run at 45°C. The binary eluting pump mobile phase on the analytical column consisted of MS-grader water at 1mM ammonium formate and 0.1% formic acid (A) and methanol 0.1% formic acid (B). The flow rate of the mobile phase was maintained constant at 0.25  $\text{mL min}^{-1}$  and the following gradient: one-minute initial hold at 5% of the organic solvent mobile phase (B) followed by a quick ramp to 10% mobile phase (B) in 0.1 min and held for 1 min. The organic solvent mobile phase (B) increased to 95% over five minutes and held for six minutes to clean the column. Finally, the column

was returned to initial chromatographic conditions and held to 5% for 1.5 min for equilibration for a total running time of 15 min.

Determination of reported limits for known analytes was performed in accordance with US EPA 2016 Revision 2 procedures with eight fortified samples and method blank samples yielding limits of detection (LODs) of 2.13, 0.79, 1.14, 2.90, and 0.58  $\text{ng L}^{-1}$  for 6PPD, 4-HDPA, 4-ADPA, 6PPD-Q, and NO-DPA, respectively. Data acquisition was accomplished by Xcalibur sequence processing, Quan Browser Version 4.4, and TraceFinder Version 5.1 software, while untargeted analysis was completed following the criteria shown on the workflow scheme in Fig. 2 and analyzed using Mass Frontier Version 8.0,  $m/z$  Vault and  $m/z$  Cloud open-source databases from Thermo Scientific. Compound detection confirmation and data processing were performed using 2022 MZmine 3 Version 3.4.16 open-source mass spectrometry data processing software. Supplemental Fig. 1 provides an example of high-resolution orbitrap mass spectra showing the presence of TWPs in extracts of the air particulate samples.

After solvent extraction, remaining particles on the filters were visually inspected using a Stemi 508 stereomicroscope equipped with an Axiocam 105 color digital camera (Carl Zeiss, Jena, Germany) and chemically analyzed by micro-attenuated total reflection-Fourier transform infrared ( $\mu$ -ATR-FTIR) spectroscopy (Fig. 3). These methods were previously described in Gao et al. (2022). Briefly, stereomicroscopy images were taken at 95 $\times$  magnification and processed by ImageJ. To estimate the abundance of TWPs, the following criteria were used to distinguish them from other particles: dark/black color, cylindrical/elongated fragment shape, rough surface texture, and a lack of any biological structures (e.g., spines or striations) (Leads and Weinstein 2019). As smaller particles are more difficult to distinguish visually, only particles with one dimension at least  $\sim$  30  $\mu\text{m}$  in size were counted. A light threshold setting of 200 was used to remove faint objects. Images are visually inspected and objects not meeting the aforementioned criteria are removed prior to counting by Image-J. We use the term *putative* TWPs, as visual observations are not sufficient to definitively discern TWPs. The TWP deposition rate was calculated based on the number of putative TWPs, the area of the separatory funnel opening (4.91  $\text{cm}^2$ ), and the time deployed (days).

To confirm the presence of TWPs on filters,  $\mu$ -ATR-FTIR was used. Black particles are not amenable to transmission and reflection modes of analyses but can provide spectra when the particle is in good contact with a  $\mu$ -ATR crystal (Gao et al. 2022). Following the visual inspection of particles by stereomicroscopy, samples were chemically characterized by  $\mu$ -ATR-FTIR using a Bruker LUMOS II microscope with a liquid nitrogen cooled MCT detector and



**Fig. 3** Stereomicroscopy image of particulate matter collected into a separatory funnel inside the Sigma-2 sampler deployed near US Highway 278 in Oxford, Mississippi (upper left). Zoomed-in optical image of putative TWPs (upper right).  $\mu$ -ATR-FTIR analysis was

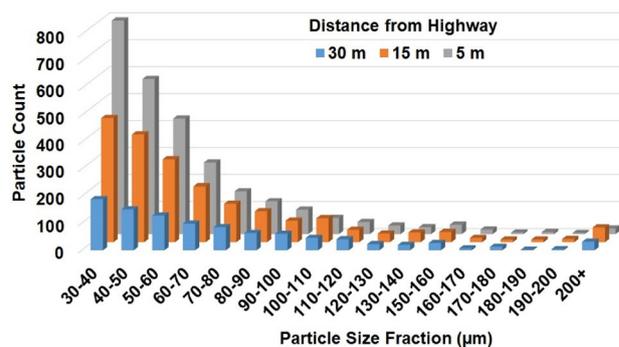
conducted on a putative TWP (remaining on the filter after extraction) at the blue and red dots. The resultant spectra (shown at the bottom) matched that of isobutylene isoprene rubber, a common synthetic elastomer in tires (Color figure online)

**Table 1** Estimated number of TWPs and their deposition rate near US Highway 278 in Oxford, Mississippi

Distance from highway (m)	TWP (n)	Deposition rate (TWPs cm <sup>-2</sup> day <sup>-1</sup> )
5	3080	63
15	2304	47
30	1107	23

a germanium ATR crystal. To facilitate analysis, particulates were transferred to a metal plate where the retractable  $\mu$ -ATR crystal could descend to contact selected particles resembling TWPs. Single spot analysis was conducted with a resolution of 4 cm<sup>-1</sup> and 12 scans using the instrument’s OPUS v8.5 software.

Unlike other microplastics, laboratory contamination with TWPs is typically not as great a concern. Nevertheless, we took precautions to minimize contamination, including preparing samples in a clean room; wearing cotton laboratory coats dyed bright orange and nitrile gloves during sample preparation; rinsing glassware with filtered (0.45 μm) water, avoiding plastic to the extent possible; and keeping samples covered with aluminum foil when not in use. We also collected and analyzed field blanks, finding no TWPs.



**Fig. 4** Particle size distribution for putative TWPs collected using the Sigma-2 sampler at varying distances from US Highway 278 in Oxford, Mississippi. Particles < 30 μm were not included in this analysis

## Results and discussion

The abundance of putative TWPs and their deposition rate increased with proximity to the road (Table 1). The majority of particles (> 90%) were < 100 μm in size (Fig. 4). The size distribution is similar to that observed by Gao et al. (2022). To confirm the presence of TWPs, spectra was collected on select particles using  $\mu$ -ATR-FTIR as described earlier. Each sample had numerous particles that yielded

**Table 2** Tire wear compounds detected in air particulate matter collected near US Highway 278 in Oxford Mississippi using the Sigma-2 passive sampler

ID	TWP compound	Formula	Category	Field blank	Distance from highway		
					5 m	15 m	30 m
4-ADPA	4-aminodiphenylamine	C <sub>12</sub> H <sub>12</sub> N <sub>2</sub>	Transformation product PPD	ND	Present	Present	Present
6PPD-Q	N-(1,3-dimethylbutyl)-N'-phenyl-p-phenyl-enediamine-quinone	C <sub>18</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub>	Transformation product PPD	ND	Present	Present	Present
1,3-DMBA	1,3-dimethylbutylamine	C <sub>6</sub> H <sub>15</sub> N	Transformation product PPD	ND	Present	< LOD	< LOD
DPG	1,3-diphenylguanidine	C <sub>13</sub> H <sub>13</sub> N <sub>3</sub>	Vehicle-related chemicals	ND	Present	Present	Present
NCBA	N-cyclohexyl-1,3-benzothiazole-2-amine	C <sub>13</sub> H <sub>16</sub> N <sub>2</sub> S	Vehicle-related chemicals	ND	Present	Present	Present
HMMM	Hexa-(methoxymethyl)melamine	C <sub>15</sub> H <sub>30</sub> N <sub>6</sub> O <sub>6</sub>	Vehicle-related chemicals	ND	Present	< LOD	Present
DCU	1,3-dicyclohexylurea	C <sub>13</sub> H <sub>24</sub> N <sub>2</sub> O	Vehicle-related chemicals	ND	Present	Present	Present
4-DBAP	4-[(1,3-dimethylbutyl)amino]	C <sub>12</sub> H <sub>19</sub> NO	Rubber tire metabolite	ND	Present	Present	Present

spectra with significant characteristics matching isobutylene isoprene rubber, a common synthetic elastomer in tires (Fig. 3). The hit quality index (HQI) comes from an algorithm that determines how well the sample spectrum matches those in the library database. For putative TWPs we observed a HQI score of > 400, which, in our experience is a good match for environmental particles. TWPs include numerous additive compounds, embedded road wear particles, and have undergone oxidation and degradation leading to spectral changes and lower HQI scores compared to pure compounds. Based on these optical and chemical analyses, we are confident that airborne PM at each site contained significant numbers of TWPs. Further we show that enough TWP mass was collected to yield a signal for several key TWP marker compounds.

The presence of both 6PPD-Q and 4-ADPA was detected in all extracts obtained from air samples collected at different distances from the highway (5 m, 15 m, and 30 m, respectively). Table 2 provides a summary of tire wear compounds identified in sample extracts. In addition, 1,3-DMBA, a transformation product, was detected in the air sample collected closest to the highway but not in samples taken at further distances. Apart from 6PPD-Q and 4-ADPA, 1,3-DMBA detected in this present study has previously been reported to exhibit toxicity toward humans (Cohen et al. 2015).

## Conclusions

The present study demonstrates that TWPs and their associated compounds, including 6PPD-Q, 4-ADPA, and 1,3-DMBA, are dispersed in the air near highways. These TWPs may present not only a hazard to aquatic organisms (through

runoff) but also to individuals inhaling them over long periods of time, and thus this route of exposure deserves further scrutiny. We also introduced a novel method combining separatory funnels with Sigma-2 passive samplers to simplify the collection and preparation of airborne PM for analyses. Future research should aim to quantify TWP compounds in air samples near roadways and the urban environment, the bioavailability of TWP compounds from inhaled TWPs, and their impact on organisms such as amphibians that have potentially significant exposure to TWPs.

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**Author Contributions** JVC conceptualized, supervised, and administered the project; BSO led the sampling campaign; BSO and MB analyzed the samples by optical and FTIR microscopy; MC analyzed the samples by mass spectrometry; MM helped with the sample preparation; and EB, MM, and VY helped with data analysis and interpretation. All authors agreed to the published version of the paper.

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## Declarations

**Conflict of interest** The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results. E.B. is the Editor-in-Chief and V.Y. and M.M. are Senior Editors for the Bulletin of Environmental Contamination and Toxicology. The entire review process was blinded within the editorial system.

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